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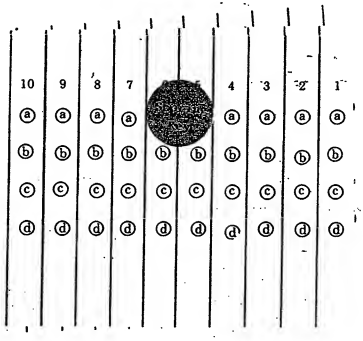
THE SMELL IDENTIFICATION TEST™ ADMINISTRATION MANUAL



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THE SMELL IDENTIFICATION TEST™ ADMINISTRATION MANUAL

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GENERAL INTRODUCTION

The purpose of this manual is twofold -- first, to provide information and normative data for valid administration and scoring of the Smell Identification Test™ (even by personnel not specifically trained in psychometric or sensory testing) and second, to review the initial studies of its development and application. Because this measuring instrument has only been recently developed, many of its potential uses have not been explored and even its most obvious applications have yet to be made. For this reason, this manual will be up-dated from time to time to include the results of more recent studies. Sensonics, Inc. would appreciate being informed of results and publications based upon the application of this test so that this information can be made available in subsequent editions of the manual.

Until the development of the Smell Identification Test™, no convenient means for quantitatively assessing smell function in a standardized manner was generally available. By incorporating microencapsulation technology and sound psychometric principles, the Smell Identification Test™ clearly filled this void. Despite the fact that the test was initially envisioned to provide only a first-step olfactory "screening" function, it became quite clear in both the clinic and laboratory that the test was much more broadly useful than initially anticipated. Indeed, it was found to be sensitive to a number of subject variables and to correlate more closely, in the clinical setting, with patients' complaints and other indices of dysfunction than measures from more traditional threshold and suprathreshold psychophysical tests. Furthermore, its high reliability has allowed it to be used in situations where previous odor identification tests were found wanting.

There is no doubt that this test has limitations in some subject groups and in some test situations, and future work will better define the limits of its applicability. For example, the test cannot be validly administered to persons with limited language ability. However, as indicated by the normative data contained in a subsequent section of the manual, it is applicable to nearly all English-speaking individuals beginning at a very young age. The test is currently being translated into several other languages and the interested investigator should contact Sensonics, Inc. for details of the release of these versions of the test. In addition, a non-verbal version of the test will soon be available for use in testing very young persons or individuals with limited language ability.

Overall, the available research data suggest the Smell Identification Test™ is highly sensitive, broadly applicable, and very useful in situations where dysfunction of the olfactory sense is present or suspected. In addition, such data indicate that it is helpful in discriminating between persons with mediocre smell function and those with a more highly developed sense of smell, as is needed in the screening of sensory panels for various industrial applications.

This manual is organized into two major sections. The first section is a review of the research work that went into the development of the test, whereas the second is a presentation of the procedures that should be followed for its valid administration.

It is absolutely essential that the test administrator be familiar with Section II, as it provides details of how the test should be administered and scored.

Although it is not absolutely necessary to read Section I of the manual to validly use the test, it is recommended that this material be read by the test administrator. A knowledge of this information will provide a better understanding of the strengths and weaknesses of the instrument which, hopefully, will translate into its most appropriate application and interpretation.

SECTION I

Development of the Smell Identification Test™

Although the initial studies describing the development of the Smell Identification Test™ are presented elsewhere [1-3], a brief overview of their procedures and findings is presented in this section. For the sake of brevity and clarity, many of the methodological and statistical details are omitted, and the interested reader is referred to the earlier publications for more specific information.

Five initial experiments, outlined in order below, led to the development of the Smell Identification Test™, although the test had as its basis an earlier prototype mentioned elsewhere [4]. In the first two experiments of this series [see 3], selection of the most appropriate stimuli was made. Subsequently, the influences of variables such as the age, gender, and ethnic background of the subjects on the scores of the developed test were examined. In the third experiment, the utility of the test in discriminating among persons with known or suspected olfactory disorders, as well as persons instructed to feign total anosmia, was established. In the fourth experiment, the instrument's test-retest reliability was determined, whereas in the fifth its scores were compared to measures derived from a traditional detection threshold procedure.

Experiment 1

Experiment 1 had four main goals. The first was to quantitatively establish, in subjects with no apparent olfactory dysfunction, psychological ratings of the perceived intensity, pleasantness, familiarity, coolness-warmth, and irritation of 50 Microfragrance samples™ of potential use in a standardized olfactory test. Such data provided basic information as to the suitability of microencapsulated odorants for testing human olfaction, as well as a basis for eliminating stimuli with problems of identifiability, irritability, or intensity from the final version of the test. The second goal was to determine whether such ratings were influenced by two means of releasing the odors from the microencapsulated crystals (scratching the surface with #120 sandpaper or with a pencil tip) and, if so, whether one means was clearly preferable to the other. The third goal was to ascertain if males and females differentially rated the stimuli (as was expected if odorants released from microencapsulated crystals behaved similarly to those from other stimulus sources; see 5-7), whereas the fourth goal was to

ascertain the relative identifiability of the 50 Microfragrance samples™ when no verbal or written cues were provided as to their identity. This information, in conjunction with that collected in the next study (Experiment 2), was utilized to eliminate stimuli that were difficult to identify.

Fifty men and women (half of each sex; mean age = 24.87 yrs, SD = 5.52 yrs) with self-reported normal smell function rated the intensity, pleasantness, coolness/warmth, irritation, and familiarity of 50 microencapsulated odorants on standard 9-point category rating scales [see 8]. The stimuli were chosen on the basis of a number of criteria, including (a) being composed of single, as well as of multiple, components (given the possiblity that the olfactory system codes information using a multiple profile/multiple receptor site process; 9), (b) spanning a number of qualitative odor classes [10], (c) evidencing (in most cases) no intranasal trigeminal stimulative properties, and (d) evidencing, in selected cases, clear-cut trigeminal stimulative ability to allow for possible detection of malingering [8].

In general, the results indicated that (a) none of the 50 odorants were perceived too extreme on any of the continua to warrent immediate exclusion from further consideration as test stimuli (Figure 1), (b) the odors differed among one another on each of the perceived attributes (Figure 1), (c) women rated the odors, on the average, as slightly more intense, more unpleasant, less cool, less irritating, and more familiar than did men, and (d) that both the sandpaper and pencil release procedures produced reasonably similar results (Figure 1). However, a few slight differences were observed between these two modes of releasing the stimuli. For example, the stimuli were rated, on the average, as slightly more familiar and less pleasant when released by sandpaper than when released by pencil. In addition, women rated stimuli released by pencil as slightly stronger (although of equal familiarity) than those released by sandpaper. Despite the fact that there was a significant tendency for the familiarity ratings to differ between the sexes as a function of the odorants evaluated, the stimuli rated as more familiar by men than by women did not differ in any obvious way from the other odorants.

Based on the findings that odors released by pencil were judged slightly less familiar than those released by sandpaper, and that women rated odors released by sandpaper stronger than odors released in this manner by men [in accord with the sex difference noted in other human olfactory work (e.g., 5-7)], sandpaper was chosen as the means for releasing the stimuli in subsequent studies.

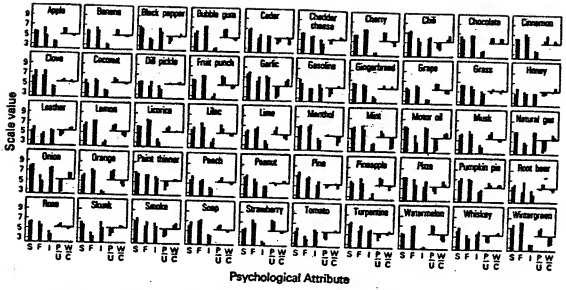


Fig. 1: Mean category ratings given to 50 microencapsulated odorant strips using #120 sandpaper (left half of each vertical bar) or the tip of a #2 lead pencil (right half of each vertical bar). S = Strength (intensity); F = Familiarity; I = Irritation; P/U = Pleasantness/Unpleasantness; W/C = Warm/Cool. For the P/U and W/C scales, the horizontal line signifies the neutral reference point. Note, for example, the marked unpleasantness and irritation ratings given to onion, but not to rose, and the coolness attributed to menthol. From [3] with permission.

Experiment 2

Experiment 2 had three main goals: First, to determine the relative identifiability of the stimulants in a forced-choice situation where alternative responses were provided; second, to omit stimuli from inclusion in the test which were not correctly identified by the majority of a large number of normal subjects; and third, to evaluate the relative influences of several subject variables, alone and in combination, upon the test scores of a large and heterogeneous group of subjects.

In the initial phase of the study (where the identifiability of the odorants was established), 1198 subjects were tested. These volunteers consisted of (a) participants of regional health fairs and public events, (b) primary and secondary public school students, (c) university students, (d) residents of homes for the elderly, and (e) employees of the Hospital of the University of Pennsylvania. Persons reporting any smell abnormalities or who were unable to correctly identify at least half of the stimulants were not included in this study group. Seventy-three percent

were White Americans and 21% Black Americans, with most of the remaining 6% not indicating their ethnicity. Sixty-two percent were female and 38% male. Eighty percent were current non-smokers, and 19% current smokers, with the remainder not reporting this information. Although a wide spectrum of ages was present in this group, disproportionately more subjects fell within the younger age ranges, as indicated by the following statistics: mean age = 35.24 (SD = 19.21); modal age = 19.0; median age = 29.29; 25th percentile = 18.88; 75 percentile = 50.3. Overall, the average ages of the two sexes, of the two major ethnic groups, and of the smokers and non-smokers were similar [see 31].

In the second phase of this study (where the influences of age, gender, race, and smoking habits upon the test scores were evaluated by multiple regression analysis), the data from most of the subjects mentioned above and from an additional number of persons (mostly elderly) were subjected to analysis. Although test scores of 1365 subjects were initially evaluated, data from 26 with apparent anosmia were omitted from the data set upon which the final regression equation was computed.

A preliminary 50-item 5-booklet Smell Identification Test™ was developed for administation in Experiment 2. In this test, which was identical in general format to the 40-item version, the 50 stimulants were presented in random order, with the exception that odors of similar psychological quality (e.g., garlic and onion) did not directly follow one another.

To aid in the selection of sets of distinct discriptors for the multiple alternative choices of each of the 50 items, the names of 51 descriptors were typed on small cards. Fifty of these names were those assigned by the manufacturer to the Microfragance samples™*, whereas one was that of "cola". These cards were then arranged by two female technicians and the author spatially on a table top with the goal of making the distances between the cards proportional to the psychological stimilarity of the odor items. For example, the garlic and onion written labels were located close to one another, whereas the chocolate and gasoline labels were placed apart from one another and at differing distances from those of onion and garlic. This simple procedure resulted in a two-dimensional space from which the three "distractor" items were selected for each odorant so as to insure their distinctiveness from one another as well as from the odorant of interest.

*Microfragrance samples™ is a registered trademark of the 3M Corporation, St. Paul, Minnesota.

In addition to sampling response alternatives relatively distinct from one another, an attempt was made to to use each of the verbal descriptors equally often and about the same number of times in the four response category positions (a,b,c & d). Although it was not possible to achieve all of these aims simultaneously, this goal was approached. Thus, 37 of the 51 descriptors appeared four times apiece, eight three times apiece, five five times apiece, one six times, and another once. No descriptor ever appeared more than twice in any of the response category positions.

As indicated in Figure 2, it was apparent that a number of the Microfragrance samples™ were poorly identified by the majority of the subjects, even though the responses were cued by written alternatives. Based on these findings and other identifiability

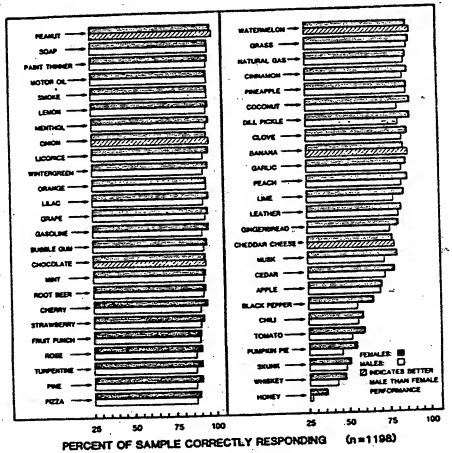


Fig. 2: Percent of subjects correctly identifying each of the 50 target microencapsulated odorants presented in a 4-alternative forced-choice response paradigm. Note that women performed better than men on most of the stimuli. From [3] with permission.

data published elsewhere [3], the following stimuli were eliminated from inclusion in the 40-item final version of the Smell Identification Test™: apple, black pepper, chili, honey, musk, pumpkin pie, skunk, tomato, and whiskey. In addition, garlic was eliminated from the final test due to its psychological similarity to onion.

To determine what influence a number of demographic variables had on the test scores (for the 40 items included in the final test), a series of multiple regression analyses were performed on data from 1339 to 1365 subjects (missing data for some variables necessitated using fewer subjects in some instances). The final regression equation fitted to subjects with Smell Identification Test™ scores 20 or greater included only variables significant at the .05 level (n = 1339):

$$Y = 33.399 + 1.055X_1 + 0.217X_2 - 0.003X_2^2 - 0.489X_3 - 1.008X_4 - 1.040X_5 - 2.172X_6 + e,$$

where $X_1 = 1$ (0) if the subject is female (male); X_2 = age of subject in years; $X_3 = 1$ (0) if the subject does (does not) currently smoke; $X_4 = 1$ (0) if the subject is (is not) nonwhite; $X_5 = 1$ (0) if the subject does (does not) report a smell problem; $X_6 = 1$ (0) if the subject does (does not) belong to an elderly sub-file (i.e., persons primarily in old-age homes who are over 65 yrs of age), and e = error term.

The R^2 value of this equation was 0.411 (SD = 3.318), and the standard errors of estimate for the seven variables were as follows: $X_1 = .188$; $X_2 = .023$; $X_2^2 = .0003$; $X_3 = .238$; $X_4 = .222$; $X_5 = .302$; and $X_6 = .525$.

Overall, these analyses indicated that gender, age, ethnic background, and smoking habits all relate significantly to scores on the Smell Identification Test™. Obviously, gender and age account for most of the variance. The relation between age, gender and Smell Identification Test™ scores is depicted in Figure 3. Note that men evidenced lower average test scores than women within nearly all age groups, and that both sexes evidenced a decrease in test performance beginning in the sixth decade of life which continued through the ninth decade.

To establish if the decrease in the scores across the older ages was due, in whole or in part, to a general decrement in memory function, the Wechsler Memory Scale (WMS, Form II) [11] was administered to 47 persons above 55 yrs of age within a day of the olfactory tests (mean age = 81.32, SD = 7.75). Because 16 of

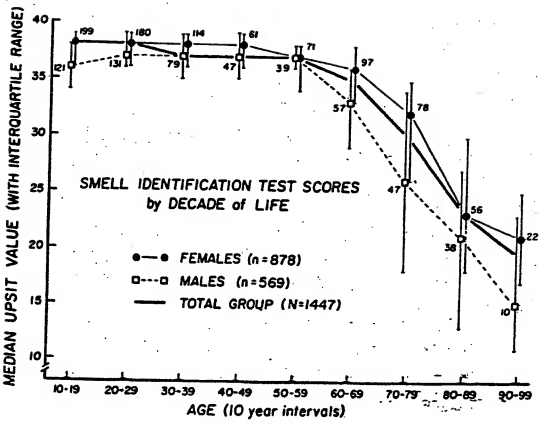


Fig. 3: Relationship between Smell Identification Test™ scores, age, and gender in a large heterogeneous group of subjects. From [3] with permission.

these individuals evidenced total anosmia, only data from those scoring 20 or above were subjected to analysis. As indicated in detail elsewhere [3], partial correlations revealed that no appreciable relationship was present between the Smell Identification Test™ scores and the WMS scores ($r = .027$, ns), despite the fact that both of these tests significantly correlated with age, per se [r (smell score, age) = $-.51$, $p <$ average Smell Identification Test™ scores with age reflects a perceptual deficit largely independent of the memory deficit measured by the Wechsler Memory Scale.

Experiment 3

The goal of Experiment 3 was to validate the Smell Identification Test™ by establishing its ability to distinguish among (a) persons with normal olfactory function, (b) persons with known or suspected olfactory dysfunction, and (c) persons instructed to feign total anosmia under the make-believe condition of receiving a large insurance payment if they successfully did so.

Five groups of subjects were administered the test:

(a) 1215 persons with normal smell function (mean age = 33.69 yrs, SD = 17.69; essentially the study population evaluated in Experiment 2 minus persons over the age of 65);

(b) 51 persons with total bilateral anosmia (mean age = 40.76 yrs, SD = 20.75); 15 had Kallmann's syndrome, with the remainder being anosmic from a number of causes [see 3]);

(c) 21 men with Korsakoff's syndrome (mean age = 57.05, SD = 8.13), an organic brain syndrome associated with a consistent pattern of lesions in the midline areas of the brainstem and diencephalon and impairment on numerous tests of olfactory function [15-17];

(d) 31 persons with multiple sclerosis (mean age = 49.03, SD = 12.56); and

(e) 158 persons with normal smell ability who were instructed to feign total anosmia under the make-believe condition that they would collect a large sum of money from an insurance company if they successfully did so. One hundred and three of these individuals had a least one year of college, whereas the remainder had a high school education or less.

As indicated in Figure 4, persons with total bilateral anosmia evidenced scores on the Smell Identification Test™ only slightly above the number expected on the basis of random responding (Mean number correct = 12.25, SD = 3.04; Median = 13). This slightly higher than chance performance was due to the inclusion of several trigeminal stimulants in the test.

Most of the Korsakoff patients evidenced aberrant scores, with a wide range in the degree of deficit being present (Mean = 15.95, SD = 7.97; Median = 14; Range = 5 - 37; Figure 4). The recent demonstration of a high correlation between scores on the Smell Identification Test™ and lumbar CSF levels of 4-methoxy-3-hydroxy-phenyl glycol (a major metabolite of norepinephrine) in a subgroup of these patients suggests that the divergent scores may reflect the degree of CNS noradrenergic pathway damage [12]. Assuming this to be the case, the Smell Identification Test™ may serve, at least in these types of patients, as a nonobtrusive index of the degree of such CNS damage.

Patients with multiple sclerosis typically scored within the normal range, although a disproportionate number fell into the

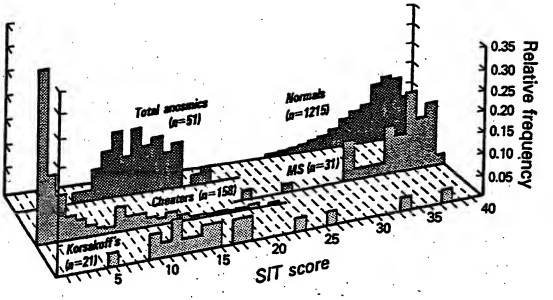


Fig. 4: Smell Identificaton Test™ scores for five groups of subjects. SIT = Smell Identification Test. See text for details. From [3] with permission.

lower section of this range, and two fell outside this range (Figure 4). A partial correlation (factoring out the effects of age, per se) revealed a weak but statistically significant relationship between the Smell Identification Test™ scores and the estimated duration of the disease ($r = -.428$, $p < .05$).

It is apparent in Figure 4 that subjects asked to feign total anosmia reported fewer correct responses than expected on the basis of random responding or than observed in persons with well-documented total anosmia. Indeed, the modal number correct in this group was zero. Overlap between the distribution of these "cheaters" with that of the total anosmics was minimal. No differences were observed between the responses of the college-educated and non-college educated subjects.

Experiment 4

A major factor which determines the usefulness and validity of a test is its reliability or stability over time; i.e., its ability to consistently measure what it is intended to measure. The purpose of Experiment 4 was to determine the test-retest reliability of the Smell Identification Test™.

Twenty-three women and 30 men (mean age = 44.13 yrs, SD = 19.98) were selected from our subject population for readministration of the Smell Identification Test™ at an interval exceeding six months from the time of the initial test. To allow for a valid computation of the test-retest reliability coefficient, we selected persons who represented the entire continuum of possible scores on the initial test. The final study group consisted of five persons with initial test scores in the 6 to 11 range, seven with scores in the 11 to 15 range, four with scores in the 16 to 20 range, thirteen with scores in the 21 to 25 range, five with scores in the 25 to 30 range, eight with scores in the 31 to 35 range, and eleven with scores in the 36 to 40 range.

As indicated in Figure 5, the scores were extremely stable despite the long interval between the two test administrations. The Pearson r between the two sets of test scores was .918 ($p <$ regression line fitted to these data (1.409) suggests the possibility that at least some of the subjects improved their performance slightly on the second test administration. Whether this slight change reflects a change in olfactoty function or simply is due to sampling artifacts requires further study.

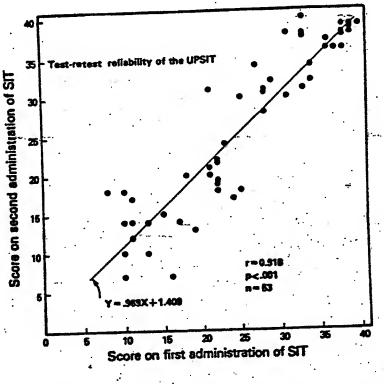


Fig. 5: Test-retest relation between Smell Identification Test™ scores in a group of subjects tested on two occassions separated by a minimum of six months. From [3] with permission.

Experiment 5

The goal of Experiment 5 was to ascertain whether scores on the Smell Identification Test™ correlate significantly with measures from a traditional odor detection threshold task. Although, theoretically, scores on a suprathreshold identification task need not correlate with detection threshold values, some degree of relationship would be expected if both tests were sampling a common domain of olfactory function.

Sixty-four men and women (mean age = 42.41; SD = 18.93) were evaluated. With the exception of six college students, these individuals were patients at the Smell and Taste Center of the Hospital of the University of Pennsylvania and evidenced varying degrees of olfactory function. Thus, a comparatively broad range of scores on both the Smell Identification Test™ and the threshold test was represented.

The subjects were administered the two tests on the same day. The threshold test was a slight modification of the forced-choice single-staircase procedure described by Ghorbanian et al [21]. A trial consisted of the presentation of two 100 ml glass sniff bottles in rapid succession in a standardized manner [see 8]. One bottle contained a given concentration of perfume-grade phenyl ethyl alcohol (a rose-like odorant relatively free of trigeminal stimulative ability) dissolved in 20 ml of propylene glycol, whereas the other contained 20 ml of propylene glycol alone. The subject indicated which of the two randomly-presented bottles evoked the stronger sensation. Even if no difference was noted, the subject was required to choose one or the other bottle. No feedback was provided as to the correctness of the responses.

The staircase was begun around the -6.0 log concentration step of a half-log step (volume/volume) dilution series extending from -6.50 to -1.00 log steps (two trials per step) and moved upwards in single log steps until correct detection occurred on two successive trials. At this point, two additional trials at that concentration level were given to decrease the likelihood of chance performance at that concentration. If a correct response did not occur on both of these trials, the staircase was moved upwards in 1.00 log steps until detection occurred on four consecutive trials at a given concentration. When correct responses occurred on all four trials, the staircase was reversed and subsequently moved up or down in 0.50 log increments or decrements, depending upon the subject's performance. Thus, the staircase was moved up 0.50 log units if an incorrect response occurred on either of the two trials, and down 0.50 log

increments if a correct response occurred on both trials. If an incorrect response occurred on the first of the two trials, the second trial was not run and a new pair of trials was begun at the appropriate next higher concentration. A minimum of 20 seconds was interposed between the pairs of trials. The geometric mean of the first four staircase reversal points following the third staircase reversal was used as the threshold measure. In cases where a subject's threshold was located outside the -6.50 to -1.00 log concentration range, the procedure of assigning the subject either the -6.50 or the -1.00 log step value, as appropriate, was adopted.

The relation between the Smell Identification Test™ scores and the detection threshold values is presented in Figure 6. Although the correlation between the two sets of measures was remarkably strong ($r = -0.89$, $p < .001$), it was likely inflated by the large number of scores of total anosmics which clustered at the lower end of the continua. When these scores (see box in Figure 6) were omitted from the computation, the correlation coefficient was -0.794 [$p < .001$].

A discussion of the findings of Section I in relation to other literature studies is presented in another publication [3].

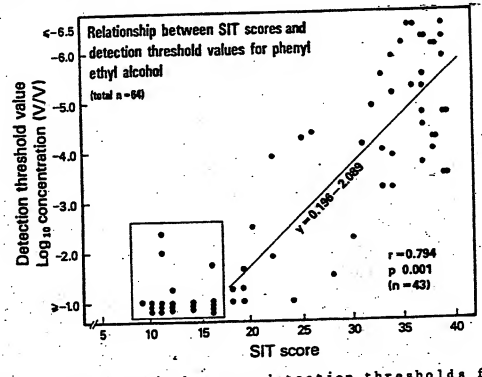


Fig. 6: Relationship between detection thresholds for phenyl ethyl alcohol and Smell Identification Test™ scores. From [3] with permission.

SECTION II

Administration and Scoring of Smell Identification Test™

Section II should be carefully read before administering the Smell Identification Test™. As with any psychometric instrument, the Smell Identification Test™ should be administered only by qualified professional personnel. Furthermore, the norms for this test and this manual should never, under any circumstances, be given to anyone not professional engaged in the scientific or medical evaluation of smell function. We recommend that this test manual and the copies of the Smell Identification Test™ be stored in a locked secure place when not in use and that, under no circumstances, the examinee be allowed to keep the test or be given direct access to its means of scoring. To insure the validity of the test, tests stored over 6 months should be evaluted by scratching the microencapsulated odor labels on a small corner section to be certain that the odors have not changed in quality.

Administration Procedures

The Smell Identification Test™ was designed to be self-administered by most literate individuals. However, care must be taken to insure that the instructions are followed exactly, and persons to whom the test is sent through the mail should be re-instructed (in a cover letter) as to the importance of providing a response to all items even if no odor is detected. In addition to failing to correctly follow instructions, some persons (particularly those with smell disorders) use the sandpaper too strongly when releasing the odorants and, essentially, sand the entire microencapsulated test strip down to the base paper. For this reason, it is best to inform the subject of this problem and to tell them to scratch the odorized surfaces by making only a few firm scrapes. For subjects being tested under supervision, it is best that the examiner or supervisor release the first odor for the subject by scratching the surface appropriately. The examiner should then indicate to the subject that this is the exact manner in which all subsequent odors should be released and that sanding is not permitted. It is essential that the subject read over the instructions before beginning the test, and that the items are sampled in chronological order. Immediately after completion of the test, the test administrator should examine it to insure that all items are complete. If not, the test should be returned to the subject immediately for completion of the

uncompleted items. Because the normative data are based upon all 40 items, incomplete booklets cannot be validly scored.

The examiner must help administer the test to persons who have impaired eyesight or who, on the basis of age or other factors, cannot read the alternatives or adequately release the odorants. In such cases, the examiner should obtain the information on the back of Booklet #1 verbally and fill it in for the subject and place the subject's name on the spaces provided on the other three booklets. The examiner should then use the sandpaper to correctly release the first odor, hold it under the subject's nose, and read aloud the response alternatives while the subject is sniffing the microencapsulated strip. In cases where the subject's eyesight is not impaired, it is permissible to allow the subject to read the alternatives as they are mentioned verbally. Finally, the examiner should mark the subject's response to each item on the columns provided on the booklet's response page. When extremely old people are tested with this instrument, it is permissible to spread the testing out over several sessions to minimize any problems associated with their attention spans or willingness to cooperate.

Because of the medical, psychological, and ethical considerations involved in assessing sensory function, it is imperative that the results of the Smell Identification Test™ be interpreted within the entire context of the individual's occupation, general health, and psychological state. For example, a score of 20 on the test is quite a different matter for a 40-year old chef than for a 40-year old sanitation worker. Likewise, as will be seen in the next section, the individual's age and gender must be taken into consideration when evaluating the test results. While a score of 20 is very abnormal for a 30 year old male (falling completely out of the range of a normal control group), such a score is not abnormal for an 80 year old male, where it would fall near the middle of the "normal" range. Assuming that the latter individual is healty, the information would be transmitted to him that while his smell ability is clearly diminished from what it was a few years before, it falls within the normal range of males within his age category.

Norms based upon the administration of the Smell Identification Test™ to 961 women and 649 men of various ages are presented in Tables 1 and 2. Despite these rather large sample sizes, it should be noted that these norms are currently being expanded and suffer from the limitation of having only a few subjects in certain age categories. For example, the numbers of children within the 4-5 yr age range is very small, necessitating caution in the interpretation of test scores from this group. The sample

of subjects upon which these norms was developed included most of the persons described in Experiment 2 of Section I of this manual. The remaining additional subjects largely consisted of more persons tested in homes for the elderly and children tested at various summer camps within the Philadelphia area. Although no claim can be made that these norms represent a truly random sample of the population at large, they represent the largest empirical collection of data on human smell function ever collected. Because of the large number of subjects examined, and because of the representation of persons from a wide range of education levels, occupations, ethnic backgrounds, and life styles, it is unlikely that these norms deviate markedly from the population as a whole. Future editions of this manual will include further breakdowns of population subgroups by factors such as occupation, ethnic background, etc., as sample sizes warrent. These norms will be updated from time to time as more "normal" individuals are evaluated.

Interpretation of a Subject's Test Score in Relation to Normative Data

The use of Tables 1 and 2 in determining an individual's percentile score is straightforward. First, the subject's total number of correct responses (maximum possible = 40) is established by use of the test's scoring key. Second, this test score is located in the far left column of Table 1 for women and Table 2 for men. The age group of the subject is then located along the top of the appropriate table and the subject's percentile score is read as the intersection of the test score row and age group column. For example, if a 47 year old female scored 35 on the test, the percentile at which she falls would be the 15th. Thus, 15% of the group of "normal" females achieved a score at or below that value, with the remainder scoring above. Such a score is clearly at the lower end of the normal group, but not markedly abnormal, as would be a score of 10 for such an individual.

In general, the following criteria have been developed for establishing a patient's olfactory diagnosis using this test instrument. With the exception of boys aged 15 yrs or less and girls aged 10 yrs or less, this classification scheme is based upon a characterization of the test scores that is independent of the age of the subjects (e.g., despite the fact that a score of 18 is in the middle of the "normal" range of scores for a 77 year old male, it is still indicative of the absolute condition of anosmia). In this classification scheme, anosmia is defined as total inability to perceive qualitative odor sensations (independent of the "common chemical sense" sensations perceived

via the trigeminal nerve), whereas microsmia is defined operationally as decreased smell ability. The term microsmia was chosen to specifically relate to the scores on the Smell Identification Test™, and does not draw a distinction between "partial anosmia" and "hyposmia" [21]. Because the operational establishment of either of these latter conditions requires both the testing of a large number of odorants and a considerable amount of effort (both of which are not practical in the clinic), the term microsmia accurately describes the condition of lessened smell function without being operationally or conceptually related to concepts which cannot be objectively confirmed in the typical clinical situation. Even if time permitted extensive threshold testing in such a context, olfactory detection threshold measures are frequently misleading, as a number of totally anosmic individuals with inflammed intranasal membranes evidence normal or supranormal detection thresholds.

SMELL IDENTIFICATION TEST SCORE	OLFACTORY DIAGNOSIS
00 - 05	Probable Malingering
06 - 19	Total Anosmia
20 - 33	Microsmia (males only)
20 - 34	Microsmia (females only)
34 - 40	Normosmia (males only)
35 - 40	Normosmia (females only)

It should again be emphasized, as indicated in Tables 1 and 2, that these criteria do not apply in the case of boys aged 15 and below and girls aged 10 and below. For these individuals, the border between normosmia and microsmia has been adjusted downward to reflect the empirical distribution of their percentiles. It should also be emphasized that the choice of these cutoff values was made on empirical grounds. Thus, in the case of the total anosmia and probable malingering categories, results from totally anosmic individuals and persons instructed to malinger revealed these values to be the appropriate cutoffs (see Fig. 4 of this manual). In addition, detailed analyses of the substances that patients reported as being detectable revealed that patients with scores below 20 noted the detection of only household substances known to have strong trigeminal stimulative properties (unpublished data). The border between microsmia and normosmia was chosen to represent a value close to the 10th percentile of adults within the middle age range.

As more individuals are added to this data base, it is conceivable that these criteria may be modified slightly. At the present time, however, they serve as relatively well-defined points and generally allow accurate categorization of smell

Table 1: Percentile Table for Females (N = 961). Numbers in body of table are percentile scores corresponding to Smell Identification Test™ scores in each age category. Numbers at bottom of table refer to sample sizes within each age category. The three solid lines crossing the columns of percentile scores represent, from bottom to top, the 25th percentile, the 50th percentile (median), and the 75th percentile, respectively. See text for details.

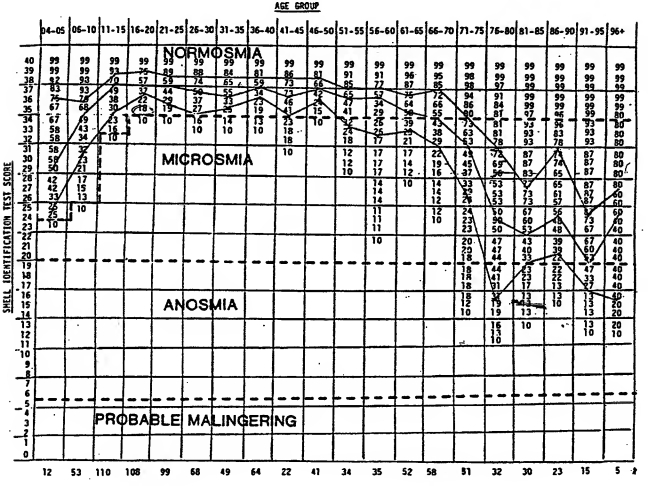
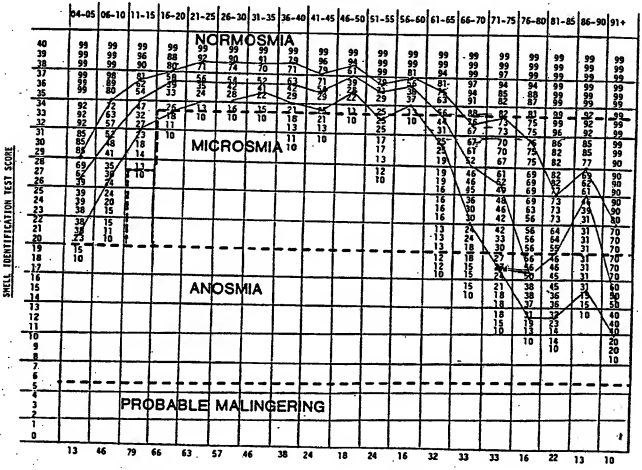


Table 2: Percentile Table for Males (N = 649). Numbers in body of table are percentile scores corresponding to Smell Identification Test™ scores in each age category. Numbers at bottom of table refer to sample sizes within each age category. The three solid lines crossing the columns of percentile scores represent, from bottom to top, the regions of the 25th percentile, the 50th percentile (median), and the 75th percentile, respectively. See text for details.



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1: J Agric Food Chem 2001 Oct;49(10):4813-7

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Aromatic profile of aqueous banana essence and banana fruit by gas chromatography-mass spectrometry (GC-MS) and gas chromatography-olfactometry (GC-O).

Jordan MJ, Tandon K, Shaw PE, Goodner KL.

U.S. Citrus and Subtropical Products Laboratory, Agricultural Research Service, U.S. Department of Agriculture, 600 Avenue S, N.W., Winter Haven, Florida 33881, USA.

Gas chromatography-mass spectrometry (GC-MS) and gas chromatography-olfactometry (GC-O) were used to determine the aromatic composition and aroma active components of commercial banana essence and fresh banana fruit paste. Totals of 43 and 26 compounds were quantified in commercial banana essence and fresh banana fruit paste, respectively. Five new components in commercial banana essence were identified as methyl butyrate, 2,3-butanediol diacetate, 2-hydroxy-3-methylethylbutyrate, 1-methylbutyl isobutyrate, and ethyl 3-hydroxyhexanoate. A total of 42 components appear to contribute to the aromatic profile in banana. Isoamyl acetate, 2-pentanol acetate, 2-methyl-1-propanol, 3-methyl-1-butanol, 3-methylbutanal, acetal, isobutyl acetate, hexanal, ethyl butyrate, 2-heptanol, and butyl butyrate had high concentrations and were most detected by GC-O panelists in the commercial banana essence. Volatile components found only in fresh banana fruit paste that were detected by aroma panelists include E-2-hexenal, limonene, and eugenol.

PMID: 11600027 [PubMed - indexed for MEDLINE]

1: J Agric Food Chem 2002 Mar 27;50(7):2016-21

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Identification and quantification of aroma-active components that contribute to the distinct malty flavor of buckwheat honey.

Zhou Q, Wintersteen CL, Cadwallader KR.

Department of Food Science and Human Nutrition, University of Illinois at Urbana-Champaign, Urbana, Illinois 61801, USA.

Characteristic aroma components of buckwheat honey were studied by combined sensory and instrumental techniques. Relative aroma intensity of individual volatile components was evaluated by aroma extract dilution analysis (AEDA) of solvent extracts and by gas chromatography-olfactometry (GCO) of decreasing headspace samples (GCO-H). Results indicated that 3-methylbutanal, 3-hydroxy-4,5-dimethyl-2(5H)-furanone (sotolon), and (E)-beta-damascenone were the most potent odorants in buckwheat honey, with 3-methylbutanal being primarily responsible for the distinct malty aroma. Other important aroma-active compounds included methylpropanal, 2,3-butanedione, phenylacetaldehyde, 3-methylbutyric acid, maltol, vanillin, methional, coumarin, and p-cresol.

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Measuring Farmstead Odors

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The nose and the brain work together to create what we perceive as odor. Our sense of smell is activated when the nose captures odor-causing chemicals, called odorants, from the air. Nerves located in the nose pass a message on to the brain when they detect an odorant. The brain then analyzes the message about the odorant.

Scientists have identified hundreds of odorants forming the collection of smells known as farmstead odors. Table 1 is a partial list of the groups of odorants released by animals and by breakdown of manure. Under each group is a list of individual chemical compounds that are commonly found in manure odors.

Odor Perception: Detection, Recognition, and Notes

The brain makes decisions about the odorant at certain concentrations. In Table 1, two numbers are listed next to the odorants; these are the detection and recognition levels for the odorants. The first number, the detection level, is the concentration in parts per billion (ppb) at which the average, healthy person first notices an odor. People cannot recognize the odor at the **detection level,** but they know they smell "something." For example, the brain notices an odor when there are 17 parts of ammonia in one billion parts of air. The second number is the **recognition** level. At this concentration, the brain begins to recognize the odorant as a distinct scent. The average human recognizes the scent of ammonia cleanser when the concentration of ammonia gas reaches 37,000 parts per billion.

Values for detection and recognition levels can be slightly difficult to comprehend. Let's look at a few examples. A person is standing on the floor of the Louisiana Superdome. The Superdome, a very large building, contains 125 million cubic feet of air space. In metric units, this equals 3.5 billion liters. Now, let's release hydrogen sulfide into the Superdome. The detection level of hydrogen sulfide given in Table 1 is 0.5 ppb. Although hydrogen sulfide is a gas, it has a weight based on the size of its molecule. A person would smell "something" if one tenth of an ounce of hydrogen sulfide were mixed in with clean air in the Superdome. The recognition level for hydrogen sulfide is 4.7 ppb. The brain would begin to recognize a faint rotten egg smell if one ounce of hydrogen sulfide was released. Skatole is a large nitrogen-containing compound largely responsible for making manure smell like manure. Skatole's detection level is 1.2 ppb, or less than one ounce of skatole in the Superdome. The recognition level of skatole is

470 ppb; therefore, the brain would not recognize a manure-like smell until about 20 pounds of skatole were released into the building.

From the previous two examples, you can see that detection and recognition levels are not always directly related. Humans can detect and recognize hydrogen sulfide at very low levels. Skatole, on the other hand, is easily detected, but only becomes recognizable at larger doses. Ammonia is an extreme example. The average human would notice "something" in the Superdome if two ounces of ammonia gas were released. They would not recognize it as ammonia until nearly 220 pounds were added!

Farm odors are never pure samples of one odorant, but rather a mixture of many different odorants. Recently, a sample of air was taken from a hog building in Germany. When chemists analyzed the sample, they measured at least 11 different organic acids, but none of the individual acids were present at detectable levels. Because the individual odorants were below the detection level does not mean they could not be smelled. The brain lumps similar odorants together as a group. You would smell something in the barn, because your sense of smell lumps all 11 organic acids together into a composite, "sour meat" smell.

Perfumers and people who blend odors call a group of odorants making a distinct scent an odor note. Let's go back to the Superdome to illustrate odor notes. If you are standing in the middle of the football field, you may not hear one person way up in the stands blowing softly into a plastic trumpet. If a hundred people blow into plastic, brass, and tin trumpets all at the same time, you will definitely hear a "note."

Odor Concentration

Farmstead odors always occur as mixtures of odorants. It is difficult and expensive to measure the concentration of each odorant in a sample. Instead, odor scientists measure the concentration of odors as a whole by grabbing a sample and presenting it to a panel of trained sniffers. The sample is diluted with odorless gas until half of the panel can no longer smell anything. When 50 percent of the sniffers can no longer detect an odor, we say the sample has been diluted to the **detection threshold**. Detection threshold is similar to detection level discussed in the previous sections. Detection threshold is the detection level of a mixture of odorants at the conditions given in the experiment.

The ratio of odorless gas to sample is called the dilution factor. **Dilution factor** is a good measure of odor concentration. The odor threshold standard used by the European Union gives odor concentration at the detection threshold the arbitrary value of one odor unit per cubic meter (OU/m^3). For example, an air sample taken from inside a dairy barn is diluted 100 times until half of the people on a panel could no longer detect an odor. The air inside the dairy barn has an odor concentration of 100 OU/m^3. Other odor threshold standards do not state odor concentration in odor units per volume. Since the dilution factor is a ratio, it has no units; therefore, the inverse of dilution factor is simply given the units OU. No matter what standard you use, the concept is the same: dilute the sample to the detection threshold, then use the inverse of dilutions as odor concentration.

Table 1. Components of Manure Odors.

Groups and Individual Odorants	Detection Level (ppb)	Recognition Level (ppb)	Odor Description
Organic Acids			

Acetic Acid	10.2	1,000	Vinegar
Propionic Acid	3.6	300	
Butyric Acid	1.1	1	Sour Meat
Iso-Valeric Acid	1.2	-	
Valeric Acid	-	20	
Alcohols, Aldehydes, Ketones			
Methanol	-	100,000	Sweet
Formaldehyde	-	1,000	Straw, pungent
Acetylaldehyde	-	210	Fruity, pungent
Acetone	4.0	100,000	Sweet, pungent
Methyl Ethyl Ketone	-	10,000	Sweet
Phenolic Compounds			
Phenol	5.7	1,000	Medicinal
p-Cresol	8.0	-	
Nitrogen Compounds			
Ammonia	17	37,000	Sharp, pungent
Methylamine	-	2.1	Fishy, pungent
Dimethlyamine	37	37	Fishy, pungent
Diethylamine	-	500	Fishy, pungent
Indole	1.0	-	Fecal
Skatole	1.2	470	Fecal, pungent
Sulfur Compounds			
Hydrogen Sulfide	0.5	4.7	Rotten Egg
Methyl Mercaptan	0.5	2.1	Rotten Cabbage
Dimethyl Sulfide	1.1	1.1	Rotten Vegetable
Diethyl Sulfide	6.0	6.0	Rotten Vegetable

Odor Character

We use the term character to describe what an odor smells like. Odor character does not change with concentration. Ammonia at 10 OU/m^3 smells the same as ammonia at 100 OU/m^3. The fourth column of Table 1 lists identifying terms used to describe the character of selected odorants. Some of the descriptive words listed in Table 1 conjure up pleasant responses. Many alcohols and ketones have "sweet" and "fruity" descriptors. Farmstead odors are mixtures of many odorants. What is pleasant by itself may be unpleasant when mixed with other compounds. In addition, unpleasant odors often add a tinge or edge to a pleasant smelling mixture. Surprisingly, indole, a nitrogen-containing compound described in Table 1 as having a "fecal" odor, is a major component in jasmine-scented perfumes.

Odor character is subjective or qualitative. In other words, it is difficult to assign a number to character. Two methods to quantify or assign a numerical value to odor character are by offensiveness and by hedonic tone.

Offensiveness

It is difficult to say exactly what farmstead odors smell like, and in the final analysis, the exact description of the smell may not matter. People know if they like the smell or not. Offensiveness is an attempt to add degrees of good and bad to "yes, it smells good - no, it smells bad" phenomena. The procedure involves three steps: an odor panel measures offensiveness; a series of samples is diluted to equal odor strength or intensity; and panelists are asked to rank the offensiveness of each sample on a scale of 0 to 5 (0 = inoffensive, 5 = strongly offensive).

Hedonic Tone

Hedonics is the science of comparisons. Odor panelists compare an unknown sample to a set of known odorants. The panel decides which of the known odorants best describe the odor. The odor is assigned a rating, called the hedonic tone, based on the comparisons. Hedonic tone provides a sense of the relative pleasantness of a sample. Pleasant odors have positive hedonic tones, and negative hedonic tones indicate unpleasant odors. Table 2 lists hedonic tones for common agricultural odors as well as some of the odorants listed in Table 1. If dead animal scent has a hedonic tone of -3.75, and rotten fruit -2.76, we can assume most people find rotting animal flesh more offensive than rotting fruit.

Table 2. Hedonic Tone of Common Agricultural Odors.

Odor	Hedonic Tone
Strawberry	2.93
Apple	2.61
Hay	1.30
Grain	0.63
Mushroom	0.52
Isovaleric Acid	-1.57
Butanoic Acid	-1.77
Mercaptans	-2.30
Ammonia	-2.47
Rotten Fruit	-2.76
Urine	-3.34
Manure	-3.36
Dead Animal	-3.75

Odor Intensity

Offensiveness tells us how bad an odor smells, and concentration gives us an idea how many molecules of odorants are floating in the air, but neither measure tells how strong an odor smells. For that, we need a third measure - odor intensity. Odor intensity is the direct measurement of a person's reaction to an odor. To measure odor intensity, scientists ask a panel to describe the strength of an unknown odor without knowing the odor concentration or dilution factor. A commonly used scale ranks intensity between 0 and 6 (0 = no odor, 6 = extremely strong odor).

Intensity experiments usually attempt to determine the relationship between concentration and intensity. An original sample of odorous gasses is mixed with clean

air in a series of dilutions and presented to a panel. Figure 1 shows the results of a series of dilutions performed on two common sources of farmstead odors - chicken house exhaust and liquid hog manure. These results demonstrate three concepts needed to understand intensity.

First, every mixture of odorants has its own relationship between concentration and intensity. Similar mixtures of odorants, such as five different samples of chicken house exhaust, have similar concentration-intensity relationships. Second, intensity and concentration are not a one-to-one relationship. If you dilute an odor sample in half, the odor intensity is not diminished by one half. In order to diminish a strong chicken house odor (I = 4) to a faint odor (I = 2), we would have to dilute a 40 OU/m^3 sample down to 5 OU/m^3. This is an 8-fold dilution.

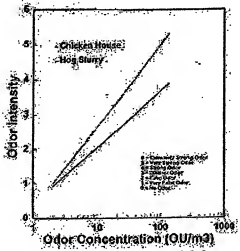

Figure 1. Relationship of Odor Intensity and Odor Concentration.

Third, intensity and character are not related. According to the results shown in Figure 1, if odor concentrations are held equal, the panel would say chicken house odor is more intense than hog slurry odor. This does not mean the chicken house smells worse than the hog slurry; it only means the chicken house smells stronger than the hog slurry. Complete description of an odor involves measuring both intensity and character. Perfumes give off high-intensity odors, but these odors are not offensive. An apple pie baking in the oven smells both strong and pleasant. Skunks release strong and offensive odors. A glass of water smells neither strong nor offensive because it has no smell at all.

Odor Persistence

Perfumers are masters in the art of blending many odors to form complex mixtures known as perfume. If one distinct odor is a note, then a mixture of many odors is a chord. Perfumers also recognize that a chord of odors can change with time. Perfumers group notes according to their relative volatility or persistence. The most persistent odors are base notes. The least persistent odors are top notes. Odorants with medium volatility are called middle notes or modifiers. When perfume is placed on the skin, the first scent smelled is the top note. Since top notes are made of volatile (or short-lived) odorants, they fade with time. Base notes remain long after the top notes have faded. Middle notes give the perfume "lift" or "body" throughout the life of the scent.

Table 3. Likely Grouping of Manure Odor Notes

Based on Relative Volatility of Odorants.

Top Notes	Middle Notes	Base Notes
Hydrogen sulfide	Aldehydes	Organic Acids
	Alcohols	Phenolic Compounds
Ammonia	Ketones	
	Amines	Indole and Skatole
	Mercaptans	Organic Sulfides (more than 5 carbons)
	Organic Sulfides (2 to 4 carbons)	Dust borne odorants

Farmstead odors are also chords of many notes. Odorants in the manure chord can be grouped in notes based on relative volatility. Table 3 classifies common manure odorants into top, middle, and base notes. Knowing that all notes do not have the same persistence can explain why the strength of farmstead odors changes over time. Consider the results shown in Table 4 from a study conducted in England. Different types of swine waste were applied 0.2 inches deep to soil inside a wind tunnel. Samples of air were collected and presented to a panel to determine odor offensiveness, concentration, and intensity immediately after spreading, and again four to six hours after application. Let us look at the results of this experiment as if the swine waste were perfume. The panel decided raw manure was definitely offensive. Odor intensity was extremely strong directly after applying raw manure, and intensity remained extremely strong six hours after application. The odors released by raw manure exposed at the soil contained persistent, strong-smelling odorants. Using perfume terminology, raw waste is heavy on the base notes.

Anaerobically digested manure paints a different picture. Anaerobically digested manure was described by the panel as faintly offensive. Initial intensity was extremely strong Das it was for the raw manure. Odors from anaerobically digested manure did not persist, however. Six hours after application, the panel smelled only faint odors in the samples. This means the anaerobically digested manure contained more top notes and less base notes. These results are consistent with the chemistry of anaerobic digestion. During digestion base notes (organic acids, skatole, and large organic sulfides) are converted to top notes (hydrogen sulfide, and ammonia) and odorless gases (carbon dioxide, methane).

Treating the raw manure by aeration reduced odors even further than anaerobic treatment. The panel described screen manure aerated at 1 to 2 mg/l dissolved oxygen as inoffensive. Why did land application odors increase with time? Why did they rise from no odor right after application to a faint odor four hours later? Aerobic bacteria are produced as raw manure is aerated, creating a large, living biomass. The aerobic biomass dies when exposed to a new environment by land application. The biomass decays anaerobically - releasing odorants similar to anaerobically digested manure.

Table 4. Odor Offensiveness, Concentration and Intensity of Land Applied Swine Wastes Based on Wind Tunnel Experiments in England.

Type of Waste	Offensiveness	Highest Measured Odor Concentration (OU/m3)		Highest Measured Odor Intensity	
		Initial	*After 4-6 hours*	*Initial*	*After 4-6 hours*
Raw Manure	Definitely Offensive	1740	320	Extremely Strong Odor	Extremely Strong Odor

Raw Manure Passed Through Screen	Definitely Offensive	250	190	Extremely Strong Odor	Extremely Strong Odor
Raw Manure Stored 14 days	Faintly Offensive	460	60	Very Strong Odor	Distinct Odor
Anaerobically Digested Manure	Faintly Offensive	350	45	Extremely Strong Odor	Faint Odor
Anaerobically Digested Manure Stored 14 days	Faintly Offensive	83	39	Strong Odor	Distinct Odor
Screened Manure Aerated at Low Dissolved Oxygen	Faintly Offensive	280	100	Distinct Odor	Faint Odor
Screened Manure Aerated at 1-2 mg/l Dissolved Oxygen	Inoffensive	60	61	No Odor	Faint Odor

Methods of Odor Measurement

Scentometer
A scentometer is a simple, hand-held odor dilution device used to measure odor concentration in the field. The person taking measurements holds the device up to his nose and breaths through the scentometer. Gases can either pass directly to the nose or pass through an activated carbon filter. The analyst chooses dilution factor by selecting the size of the hole passing unfiltered air. Advantages of the scentometer are its portabilty, its simplicity of use, and its ability to give immediate values for odor concentration and intensity. It is particularly useful for measuring intensity of odor sources. The main disadvantage is that it is difficult to overcome the analyst's personal bias in measurement. Also, the analyst's ability to distinguish odors diminishes the longer he is exposed to odors. Scentometer readings taken after an hour of sniffing may vary from readings taken when first arriving at the farm.

Olfactometer
An olfactometer is a laboratory device that distributes sample dilutions to odor panelists. A sample is collected at the farm and stored in a teflon or kevlar bag and brought into the laboratory. There are a number of variations on the olfactometer, but all devices do the same thing: the original sample is diluted with a stream of odorless gas and presented to a sniffer. Olfactometry is used to measure odor concentration, intensity, and offensiveness. Flexibility of use is the main advantage. Disadvantages are expense of operation and difficulty collecting representative samples of odorous gas.

Electronic Nose
Electronic noses mimic the human olfactory system using polymer sensors to simulate receptors in the nose and a computer to simulate the brain. Chemical composition of sensors is altered so each sensor responds differently to a given odorant. The main use of an electronic nose is to compare differences between mixtures of odors. The primary drawback is the electronic nose must "learn" a pattern of sensor responses before it can make future comparisons. Work is underway to devise electronics that will allow the nose to "guess" new odors. If properly trained, electronic noses may prove valuable in measuring odor character. A second drawback is, at the current level of technology; electronic noses are not sensitive at low odor concentrations.

Chemical Methods
Chemical methods are used to determine the actual concentration of individual odorants in a sample taken from the field. The most common instrument used in odorant analysis

is a gas chromatograph with a mass spectrometer detector. Like the electronic nose, a gas chromatograph distinguishes compounds by comparing to a reference standard. The main drawback to chemical methods is the shear number of potential odorants needed to analyze in a single sample of farmstead odors.

F-1740, Measuring Farmstead Odors *(pdf file)*

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KIRK-OTHMER

CONCISE ENCYCLOPEDIA OF CHEMICAL TECHNOLOGY

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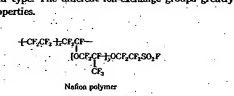
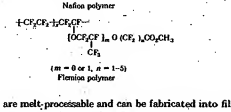
new electrolytic process for chlor-alkali production using perfluorinated ion-exchange membranes (see Alkali and chlorine products). Flemion is a carboxylic acid type. The different ion-exchange groups greatly affect membrane properties.



Nafion polymer



(m = 0 or 1, n = 1–5)
Flemion polymer

Both polymers are melt-processable and can be fabricated into films by extrusion-molding. These films can be easily converted to the corresponding ion-exchange membrane by alkaline hydrolysis.

Preparation

The general procedure includes synthesis of a perfluorovinyl ether moiety with a functional group, its copolymerization with tetrafluoroethylene in the presence of a radical initiator in an aqueous or inert organic medium, and the formation of a membrane.

Fabrication. The crystallinity of the copolymer depends upon the content of the functional comonomer. Amorphous or partly crystalline copolymers are fabricated into films (100–250 μm thick) with conventional extrusion techniques. The films are usually reinforced with Teflon cloth and converted to sulfonic- or carboxylic-acid-type ion-exchange membranes by alkaline hydrolysis.

A sulfonic acid group can be converted to a carboxylic acid group:

$$\cdots\text{—O}(\text{CF}_2)_n\text{SO}_2\text{X} \rightarrow \rightarrow \cdots\text{—O}(\text{CF}_2)_{n-1}\text{CO}_2\text{H}$$
$$(\text{X} = \text{Cl or F})$$

The sulfonyl halide group is converted to sulfinic acid by reduction and then the carboxylic acid group, having one CF_2 less than the original chain of sulfonic acid, is formed through a desulfonylation reaction.

Applications

In the electrolysis of brine, a cation-exchange membrane is used. DuPont has developed a variety of Nafion series. The Nafion 300 series produces 10–20% caustic soda. For the production of 20–28% caustic soda, the Nafion 200 series was developed. The Nafion 900 series membranes are carboxylate–sulfonate two-layer membranes with ca 95% current efficiency at 33% caustic soda.

Asahi Glass has developed the Flemion series. For the production of 35% caustic soda, a standard Flemion 230 is used advantageously with a current efficiency of 94%. With the Flemion 700 series, gas bubbles can be removed easily from the membrane surfaces.

A new electrolytic process with a zero-gap cell, called the AZEC system, combined with Flemion 722 or 753 and a new electrode system, results in drastic reductions in energy consumption.

Asahi Chemical Tokuyama Soda improved the electrolytic performance of Nafion-type membranes by chemical modification of the anode-side surface of the carboxylic acid-type membrane.

MASAAKI YAMABE
Asahi Glass Company, Ltd.

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PERFLUORO COMPOUNDS. See Fluorine compounds, organic.

PERFUMES

Perfumery is the art of producing fragrances through the combination of odoriferous substances. The word perfume is derived from the Latin meaning "through smoke". Throughout history, perfumes have played an important role in human lives, and have been associated with notions of happiness, beauty, and satisfaction. Until this century, fragrance materials have been derived from natural sources, which has placed limitations on odor types and markets (see also Cosmetics; Odor modification). The increased use of perfumes in the last thirty years would have been impossible without the development of the chemistry that allowed the invention of totally new odoriferous molecules as well as the synthesis of natural ones. Fragrances are no longer a luxury for the rich but today are incorporated routinely in a great number of products that are in daily use.

Fragrance Raw Materials

Natural products. Essential oils are volatile materials produced from odorous plant material, generally by water or steam distillation or by expressing (see Oils, essential).

A concrete is an extraction almost exclusively from vegetable origin such as leaves, bark, flowers, and fruit. This is normally obtained by extraction with hydrocarbon solvents.

Absolutes are the alcohol-soluble portion of concretes, obtained by extracting the concretes with alcohol. Resinoids are perfume materials obtained by extraction of plant resinous substances with hydrocarbon solvents.

Tinctures are alcoholic solutions. In perfumery, these are generally the solutions obtained by maceration of various odorous materials with alcohol.

Natural products used in perfume include anabergis, benzoin, castoreum, civet, clove leaf oil, galbanum, jasmine absolute, labdanum, maté, melilot, mimosa, musk tonquin, myrrh, oakmoss or mousse de chêne, olibanum, opoponax, orris, patchouli, rosemary oil, sandalwood oil, vetivert oil, and violet leaves absolute.

Aroma Chemicals

During the last 20 years, there has been a rapid advance in the capabilities of instrumental techniques for the separation and identification of volatile organic substances. Of particular importance to the perfumery industry was the development of capillary gas chromatography columns and the ability to use them directly in tandem with a mass spectrometer. Computer technology is used to interpret the vast amount of data generated by such a combination of instruments. These developments along with Fourier transform nmr spectroscopy have allowed discovery and identification of extremely minute odoriferous samples and have revolutionized not only the analysis of essential oils and extractives but also the direction of the synthesis of aroma chemicals.

Research in aroma chemicals can be divided into three general categories: (*1*) duplication of naturally occurring chemicals, for example, phenethyl alcohol, which occurs in rose oil; (*2*) chemical modification of abundant, naturally occurring materials, eg, acetylated vetivert oil ("vetivert acetate") from vetivert oil, and vanillin (qv) from lignin (qv); and (*3*) synthesis based on industrial organic feedstocks, eg, nitro musks.

Aroma chemicals are usually cheap and available in any needed quantity (see also Alcohols, higher aliphatic; Aldehydes; Benzaldehyde; Benzoic acid; Cinnamic acid; Cinnamaldehyde; Cinnamyl alcohol; Coumarin; Esters, organic; Indole; Ketones; Salicyclic acid and related compounds; Terpenoids; Vanillin).

Odor Vocabulary

The descriptions and groups of fragrance raw materials are helpful in evaluating existing aroma chemicals or newly developed materials. To illustrate the use of the odor vocabulary, two well-known materials are

United States Patent [19]

Meador et al.

[11] **Patent Number: 5,031,764**

[45] **Date of Patent: Jul. 16, 1991**

"K"

[54] **APPARATUS FOR DESIGNING PERSONALIZED PERFUME**

[76] Inventors: **Clifford A. Meador**, 1070 Mission Lyn Rd., Santa Barbara, Calif. 93105; **David S. Noble**, 1422 "D" Banner Ave., Summerland, Calif. 93067

[21] Appl. No.: **394,529**

[22] Filed: **Aug. 16, 1989**

[51] **Int. Cl.**[5] **A45D 34/00**; B65D 85/72; B65D 73/00

[52] **U.S. Cl.** **206/232**; 206/459; 206/456; 206/484; 206/820; 206/823; 206/549

[58] **Field of Search** 206/459, 449, 456, 484, 206/820, 823, 824, 831, 549, 568, 569, 542, 545, 223, 232; 73/864.72, 864.91

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Primary Examiner—Tom Noland

Attorney, Agent, or Firm—Fleit, Jacobson, Cohn, Price, Holman & Stern

[57] **ABSTRACT**

A plurality of tapered sample strips are secured at one end to a border strip to faciliate the performance of perfumery by a relative novice in the field. The tapered sample strips include the fragrance of a note, which is an essential oil, used in the perfume industry to manufacture perfumes. The series of tapered sample strips secured to the border strip are prepared for sampling by first separating two sets of border strips with their associated tapered sample strips from a die-cut sample sheet of heavy paper. The identifying indicia and color for each fragrance is clearly indicated. A tiered sample rack or case, including a plurality of capped bottles, corresponding to the number of tapered sample strips on each border strip, contains different notes or essential oils which correspond in order to the marked indicia on the tapered sample strips. Each tier of bottles corresponds to a separate border strip and its associated tapered sample strips.

14 Claims, 3 Drawing Sheets

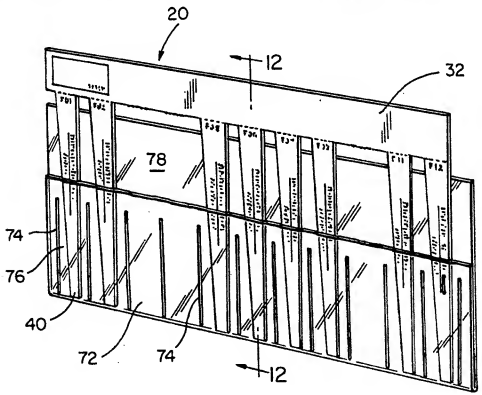


FIG. 1

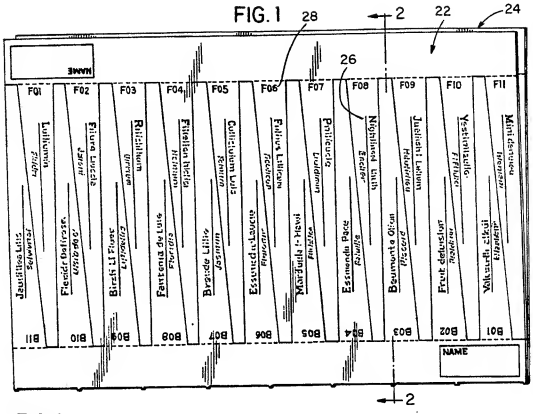


FIG. 2

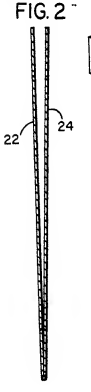


FIG. 3

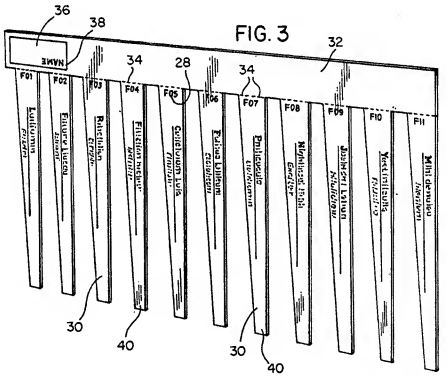


FIG. 4

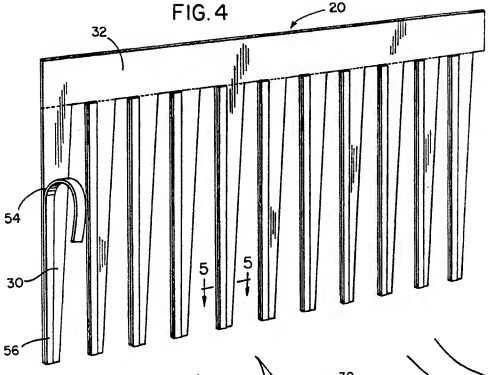


FIG. 5

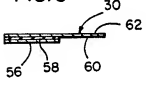


FIG. 6

FIG. 7

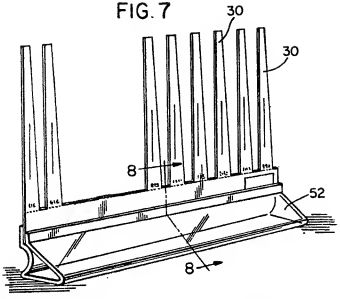


FIG. 8

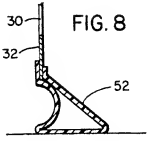


FIG. 9

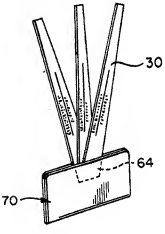


FIG. 10

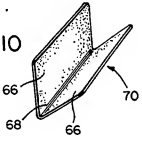


FIG. 12

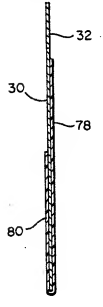
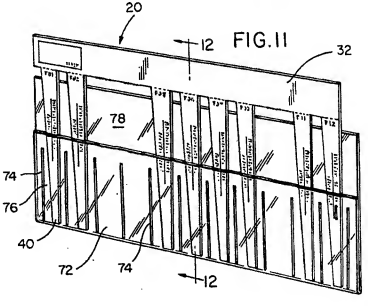


FIG. 11



APPARATUS FOR DESIGNING PERSONALIZED PERFUME

FIELD OF THE INVENTION

This invention relates to a method and apparatus for producing custom perfumes based on an individual's taste. By the invention, an individual may select a combination of fragrances which are desired to be incorporated into a personal perfume.

BACKGROUND OF THE INVENTION

Prior to the creation of the present invention, the science of perfumery was a lengthy and involved process for selecting a desired fragrance for a perfume. Typically, unlabelled scented sticks were sampled and a judgment made as to how to vary the scents to achieve a desired scent. The involvement of the process made the selection of a perfume unavailable to the average person.

SUMMARY OF THE INVENTION

By the present invention, the disadvantages of the prior art have been overcome. An individual is now capable of designing a custom fragrance in a short time span, enabling the average person to create his or her own perfume or cologne.

By the present invention, a plurality of tapered sample strips are secured at one end to a border strip to facilitate the performance of perfumery by a relative novice in the field. The tapered sample strips include the fragrance of a note, which is an essential oil, used in the perfume industry to manufacture perfumes.

The tapered configuration of the sample strips allows for ease of handling during the perfume creation phase when the customer is sampling different combinations of the various fragrances. The broader base of the sample strip allows the customer to hold the sample strips as he/she would handle playing cards. Thusly arraying them in a fanlike fashion in one hand, allowing for the evaluation of their combined fragrance by fanning them in a sideways fashion under their nose.

The tapered sample strips secured to the border strip are classified into family of notes by color of the tapered sample strips, which are grouped together by the same family having the same color strip. An example of the different fragrance families are fruit, floral, fantasy, herbal, oriental/spice, fougere, chypre/wood, and leather. It is therefore easy to distinguish between the different families of notes. The individual tapered sample strips are further identified by indicia of name and number and arranged by family in an order that is optimum for sampling. The sample strips are arranged in order such that they are sampled from lightest to heaviest in fragrance. The lighter fragrances evaporate quickly, while the heavier fragrances evaporate more slowly. It is vital to the perfuming process to smell less distinctive, lighter fragrances first and the more distinctive, heavier fragrances last. This allows the olfactory glands to obtain a true scent of each fragrance.

The series of tapered sample strips secured to the border strip are prepared for sampling by first separating two sets of border strips with their associated tapered sample strips from a die-cut sample sheet of heavy paper. The identifying indicia and color for each fragrance is clearly indicated. Typically, four border strips, each having eleven tapered sample strips are formed from two sample sheets. However, the number of tapered sample strips may be increased or decreased according to the number of fragrances to be used in the sampling process.

A tiered sample rack or case, including a plurality of capped bottles, corresponding to the number of tapered sample strips on each border strip, contains different notes or essential oils which correspond in order to the marked indicia on the tapered sample strips. Each tier of bottles corresponds to a separate border strip and its associated tapered sample strips.

The caps are removed from the bottles in a first row located on a tier of the sample case. The border strip with the corresponding tapered sample strips are lowered into the bottle so that the ends of the tapered sample strips just touch the essential oil in each bottle. When the tapered sample strips have been dipped in the corresponding essential oils, the border strip is temporarily stored in a holding rack. This process is repeated for each of the three other border strips and their associated tapered sample strips for the remaining three tiers and associated three rows of capped bottles containing essential oils.

The sample strips are detached from the border and stored in the holding rack until a customer is given each of the now fragranced tapered sample strips. Alternately, the tapered sample strips may include a peel-off strip for release of a microencapsulated fragrance.

Fragrances are microencapsulated in a special polymer. The microencapsulated fragrance is then applied by a spray or other means to a backing sheet with an adhesive vehicle. A sheet of paper covers the microencapsulated fragrance. The adhesive vehicle containing the microencapsulated fragrance is then allowed to dry. When the paper, such as a peel strip, is separated from the backing sheet, the microcapsules open, thus freeing the fragrance into the air.

After the customer has sampled each of the fragrances of the tapered sample strips by passing the strip by their nose, the customer is now able to create an individualized perfume. The customer is then given a set of the four border strips and associated tapered sample strips and the less desirable fragrances of labelled tapered sample strips are removed from the border strip. Then, only the tapered sample strips containing desirable fragrances are dipped into the corresponding tier of the sample case with the corresponding bottles of essential oils. Since the less desirable sample strips have been removed, only the desired essential oils will be transferred to the remaining tapered sample strips.

The customer, then, only dealing with the most desirable fragrances, sniffs and selects the combination of tapered sample strips having the desired combined fragrance for their personalized perfume. The selected tapered sample strips are secured together so as to clearly identify the desired fragrances which should be combined to produce their own custom perfume. Based upon the selected fragrances, a customized perfume is then made based on an individual's personal taste.

It is therefore an object of the present invention to provide a method and apparatus for sampling fragrances of essential oils.

It is another object of the present invention to sample the fragrances of essential oils by dipping a plurality of spaced tapered sample strips secured to a border strip with a free end of the tapered sample strip being immersed in bottles of essential oils corresponding to indicia and color of the tapered sample strips.

It is another object of the present invention to sample the fragrances of different essential oils and select certain fragrances which are provided on another border strip having tapered sample strips corresponding only to the selected preferred fragrances so as to sample a reduced number of fragrances of essential oils.

It is still yet another object of the present invention to sample the fragrances of different essential oils and select certain fragrances which are provided on another border strip having tapered sample strips corresponding onlY to the selected preferred fragrances so as to sample a reduced number of fragrances of essential oils and then select only the desired fragrances to be combined in a customized perfume.

These and other objects of the invention, as well as many of the intended advantages thereof, will become more readily apparent when reference is made to the following description taken in conjunction with the accompanying drawings.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. **1** is a side elevation view of two die-cut sample sheets having preprinted tapered sample strips.

FIG. **2** is a sectional view taken along line **2—2** of FIG. **1**.

FIG. **3** is a perspective view of a single border strip with associated tapered sample strips.

FIG. **4** is a perspective view of an alternate embodiment of tapered sample strips secured to a border strip.

FIG. **5** is a sectional view taken along line **5—5** of FIG. **4**.

FIG. **6** illustrates the dipping of a border strip and its associated tapered sample strips into a row of bottles containing essential oils with the bottles being located on a tier of a sample case.

FIG. **7** illustrates a holder for a border strip having selected tapered sample strips.

FIG. **8** is a sectional view taken through line **8—8** of FIG. **7**.

FIG. **9** illustrates a securing card for sealing selected tapered sample strips together.

FIG. **10** illustrates the secured card in an open condition.

FIG. **11** illustrates a chambered shipping holder with inserted tapered sample strips.

FIG. **12** is a sectional view taken along **12—12** of FIG. **11**.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

In describing a preferred embodiment of the invention illustrated in the drawings, specific terminology will be resorted to for the sake of clarity. However, the invention is not intended to be limited to the specific terms so selected, and it is to be understood that each specific term includes all technical equivalents which operate in a similar manner to accomplish a similar purpose.

With reference to the drawings, in general, and to FIGS. **1** through **4**, in particular, a fragrance sampling card embodying the teachings of the subject invention is generally designated as **20**. With reference to its orientation in FIG. **1**, the sampling reference card is shown in its die-punched condition prior to separation from two sample sheets **22** and **24**. Each sample sheet is printed to include the identifying indicia of name **26** and number **28**.

Each sheet **22** and **24** is separated from each other and then each sheet is separated to include two sample cards **20** which include a plurality of tapered sample strips **30** connected to a border strip **32** by perforated score lines **34**. The border strip **32** includes a box **36**, with indicia **38**, for prompting entry of the name of a customer.

In the sample card **20**, shown in FIG. **3**, eleven tapered sample strips are associated with border **32**. The number of tapered sample strips may be greater or less so as to correspond to a number of bottles of essential oils to which the free ends **40** of the strips **30** are to be dipped. In FIG. **3**, for example, seven of the sample strips including the identification FO1 through F07 may be of the same color whereas strips FO8 and FO9 may be of a different color and strips F10 and F11 would be of a third color. The use of different color strips identifies groups of families of fragrances and to group the fragrances of a particular family together.

In FIG. **6**, a sample case **42** made of transparent plastic, includes four steps or tiers **44** for supporting a row of bottles **46**, having caps **48**, for securing essential oils **50**, with a different essential oil in each bottle. The number of bottles **46** on each tier **44** of transparent case **42** corresponds to the number of tapered sample strips connected to a border strip.

Initially, a sample sheet **20** is grabbed by the border strip **32** and free ends **40** of the tapered sample strips are dipped into the essential oils contained in the bottles which correspond to the labeling indicia of the tapered sample strip. The strips are tapered so that the free end of the strips will fit within the opening of the bottle **50** and the wider opposite end is easily held in the hand of a customer in a fan-like array.

The sample card is then inverted from the position shown in FIG. **6** and placed within a rack **52** in a position similar to that shown for the sample sheet shown in FIG. **7**. However, it is noted that the sample sheet shown in FIG. **7** is missing three of the tapered sample strips, which will be explained later. After the essential oils have set in the free end of the strips of the sample card, the sample card is ready for sampling by the customer.

As an alternate method of transferring a different fragrance to each of the tapered sample strips, the sample sheet **20** shown in FIG. **4** may be used, which is identical on its front face to the sample sheet shown in FIG. **3**. On the rear face which is shown in FIG. **4**, a strip of a microencapsulated fragrance is located along an edge **54** of the tapered sample strip **30**. The edge **54** is covered by a removable peel strip **56** which upon removal, releases a fragrance at an area along the edge **54** of the strip **30** by breaking of the capsules containing the fragrance. As shown in more detail in FIG. **5**, a scented area **58** is located sandwiched between the rear surface **60** of the sample strip **30**, opposite the surface **62**, which includes the printed indicia and color for a particular fragrance and its family, and the peel strip **56**.

By the method of FIG. **6** or that disclosed in FIGS. **4** and **5**, the result is a border strip with a plurality of tapered sample strips having a different fragrance on each strip as identified by labelling indicia including color, title and reference number. After the scenting of the tapered sample strips has been accomplished the scent card **20** is ready for sampling by a customer and ultimate selection of a personalized perfume.

The four sample cards produced from the two sample sheets **22** and **24** are each dipped in a different row of essential oils contained in the sample case **42** or include

a microencapsulated fragrance so as to provide, in this example, 44 different fragrances of essential oils which are identified by number, title and family.

To sample the different fragrances, the tapered sample strips 30 are removed from the border strip 32 along the perforation lines 34. The individual sample strips are either held within rack 52 or passed directly to a customer to allow the customer to sample an individual fragrance. In the dipped sample strip the fragrance is sniffed whereas in the microencapsulated fragrance strips, the peel strip is removed to release a fragrance and then sniffed.

The customer notes the most desired fragrances by fragrance name or identifying number. If a particular fragrance is enjoyed, other fragrances of the same family are easily identified by the color coding of fragrances belonging to the same family. After each of the individual tapered sample strips have been sampled, each customer is given a complete sample card 20. The customer then removes the tapered sample strips of the fragrances they have identified as not being of particular interest to them. Only the tapered sample strips identifying fragrances they would consider ultimately to use in their personalized perfume then remain on the border strips of the sample cards. The name of the customer may be inserted into the box 36 for proper identification.

Following the procedure previously explained for the dipping of the sample cards, the sample cards, with the tapered sample strips with the less desirous fragrances having been removed by the customer, is then dipped into the corresponding row of essential oil bottles in the sample case. The sample card is then placed into the rack 52 as shown in FIG. 7, which also shows a typical sample card with some of the tapered sample strips having been removed for saturation of the tapered sample strips by the essential oils.

When the customer is ready to sample the dipped sample strips, the sample card with the selected sample strips is returned to the customer in the rack or first removed from the rack and then returned to the customer. The customer then resamples the selected fragrances by sniffing. The selected sample strips may be removed from the sample sheet by ripping along the perforation lines 34 and holding several sample strips together to obtain a combined potential fragrance for a personalized perfume.

After mixing and matching the most desirous fragrances, a decision is ultimately made as to a desired personalized perfume for a customer. The selected finalist strips are placed with their perforated strip ends 64 between the sides 66 of a prepasted or adhesive layer and folded about fold line 68 to secure the desired sample strips 30 in a case 70, as shown in FIG. 9, to hold the finally selected sample strips 30. Based upon the selected sample strips, a personalized perfume is mixed for a particular customer.

Sampling of the different fragrances of the essential oils may also be accomplished by shipping the sample cards 20 within a transparent shipping case 72, which includes separation lines 74, so as to form individualized chambers 76 to contain a fragrance without mixing with adjacent fragrances, between a rear sheet 78 and a front sheet 80, so as to seal the fragrance located at a free end 40 of a tapered sample strip 30.

Having described the invention, many modifications thereto will become apparent to those skilled in the art to which it pertains without deviation from the spirit of the invention as defined by the scope of the appended claims.

We claim:

1. A sample sheet kit for sampling different fragrances of essential oils, said sample sheet comprising
- a plurality of elongated sample strips having two ends,
- indicia means located on said sample strips for identifying a fragrance of a particular essential oil with a different fragrance being identified on each of said elongated sample strips,
- one end of said plurality of elongated sample strips being scented with a fragrance corresponding to a fragrance identified by said indicia means,
- a case formed by a front sheet and a rear sheet secured together, said one end of said plurality of elongated sample strips being sealed between said front sheet and said rear sheet to avoid mixing of a particular fragrance of one elongated sample strip with an adjacent elongated sample strip,
- the other end of said elongated sample strips being holdable in a hand of a customer in a fan-like array so that upon sampling of said sample strips, a determination is made of a combination of desired fragrances held in the hand of the customer as represented by at least two sample strips which are combined according to the essential oils identified by the indicia means to produce a customized perfume.

2. A sample sheet as claimed in claim 1, wherein said sample strips are tapered inwardly from a border strip to said one end.

3. A sample sheet as claimed in claim 1, wherein said indicia means identifies a family common to at least two of said sample strips.

4. A sample sheet as claimed in claim 1, wherein said sample strips are spaced from each other along a length of a border strip.

5. A sample sheet as claimed in claim 1, wherein said sample strips are removably secured to a border strip.

6. A kit for selecting fragrances for a personalized perfume, said kit comprising:
- a plurality of sample sheets each including a border strip, a plurality of sample strips connected at one end to said border strip, and indicia means located on said sample strip for identifying a particular fragrance,
- a sample case, and
- container means located in said sample case for containing an essential oil, said container means being aligned in said case to correspond with a spacing of said sample strips so that a free end of said sample strips when dipped into said container means contacts an essential oil contained in said container means having a fragrance corresponding to the particular fragrance identified by said indicia means of said sample strip.

7. A kit for selecting fragrances as claimed in claim 6, wherein said sample case includes tiered shelves for supporting rows of said container means.

8. A kit for selecting fragrances as claimed in claim 6, wherein said sample strips are removably connected to said border strip by perforations.

9. A kit for selecting fragrances as claimed in claim 6, wherein said indicia means identifies a family common to at least two of said sample strips.

10. A kit for selecting fragrances as claimed in claim 9, wherein said sample strips are tapered inwardly from said border strip to said free end.

11. A system for sampling different fragrances of essential oils for selecting preferred fragrances for combination in a customized perfume, said system comprising:

a plurality of elongated sample strips having two ends,

indicia means located on said sample strips for identifying a fragrance of a particular essential oil with a different fragrance being identified on each of said elongated sample strips,

fragrance means located at one end of each of said elongated sample strips for storing a different fragrance on each sample strip corresponding to the particular fragrance identified by said indicia means,

a case formed by a front sheet and a rear sheet sealed together, said plurality of elongated sample strips being sealed between said front sheet and said rear sheet to avoid mixing of a particular fragrance of one elongated sample strip with an adjacent elongated sample strip,

the other end of said elongated sample strips being held in a hand of a customer in a fan-like array so that upon sampling of said sample strips, a determination is made of a combination of desired fragrance held in the hand of the customer as represented by at least two sample strips which are combined according to the essential oils identified by the indicia means to produce a customized perfume.

12. A system for sampling different fragrances as claimed in claim 11, wherein said fragrance means includes a microencapsulated fragrance released by removal of a peel strip.

13. A system for sampling different fragrances as claimed in claim 12, wherein said indicia means include a title of a particular fragrance.

14. A system for sampling different fragrances as claimed in claim 12, wherein said indicia means include a color representative of a particular family of fragrances.

* * * * *

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(12) **United States Patent**
Sunshine

(10) **Patent No.: US 6,606,566 B1**
(45) **Date of Patent: Aug. 12, 2003**

(54) **COMPUTER CODE FOR PORTABLE SENSING**

(76) Inventor: **Steven A. Sunshine**, 985 S. Oakland Ave., Pasadena, CA (US) 91106

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 31 days.

(21) Appl. No.: **09/703,467**

(22) Filed: **Oct. 31, 2000**

Related U.S. Application Data

(60) Provisional application No. 60/188,307, filed on Mar. 10, 2000, provisional application No. 60/164,022, filed on Nov. 4, 1999, and provisional application No. 60/162,683, filed on Nov. 1, 1999.

(51) **Int. Cl.**[7] **G06F 13/00**

(52) **U.S. Cl.** **702/22**; 702/116; 702/120; 702/123

(58) **Field of Search** **702/22**, 116, 120, 702/123; 600/345; 379/88.22

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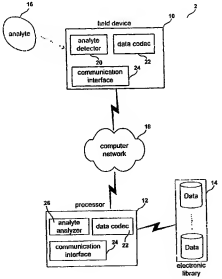
Assistant Examiner—Felix Suarez
(74) *Attorney, Agent, or Firm*—Townsend and Townsend and Crew, LLP; Horace H. Ng

(57) **ABSTRACT**

The present invention relates to a computer program product or code in memory for detecting and transmitting sensory data from a portable field device **10** to a processor **12** via a computer network **10** for analytic purposes. The product includes a code directed to capturing analyte data pertaining to an unknown analyte using a field device **10**. The product further includes a code directed to encoding the captured analyte data and transmitting the encoded analyte data via a computer network **18** to a processor **12** for analysis. The product also includes a code directed to performing an analysis of the captured analyte data at a remote location by the processor **12** using data of known analytes retrieved from an electronic library **14**. This code and others can be used with the present invention to perform the functionality described herein as well as others.

33 Claims, 8 Drawing Sheets



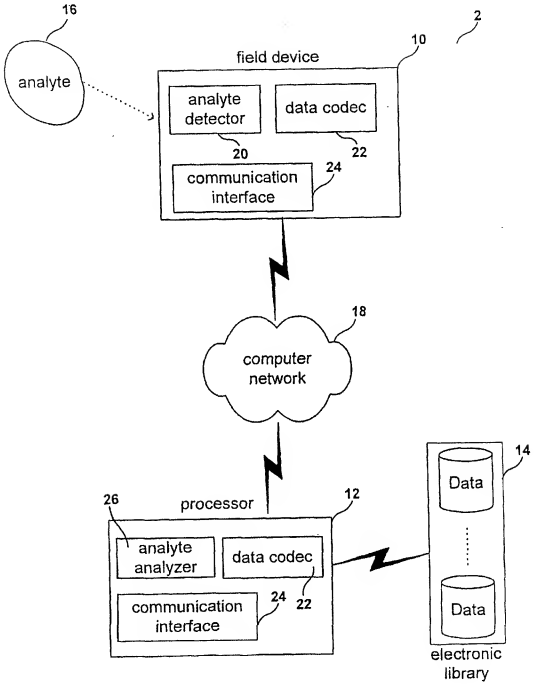

16
analyte
2
10
field device
analyte detector
data codec
20
22
communication interface
24
18
computer network
14
Data
Data
26
processor
12
analyte analyzer
data codec
24
22
communication interface
electronic library

Fig. 1

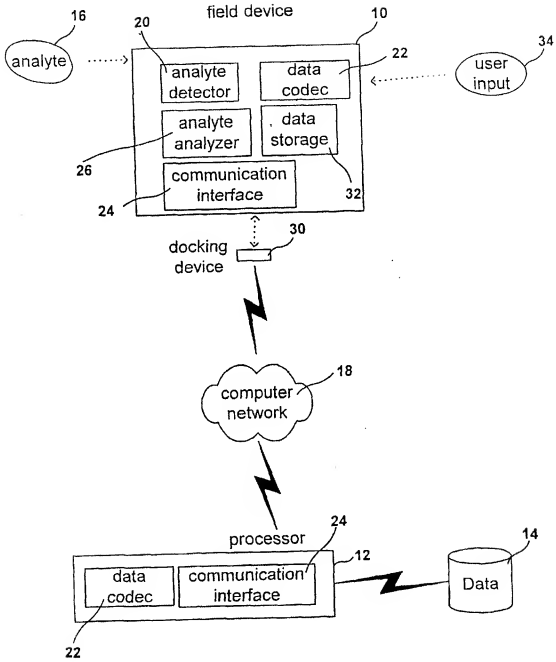
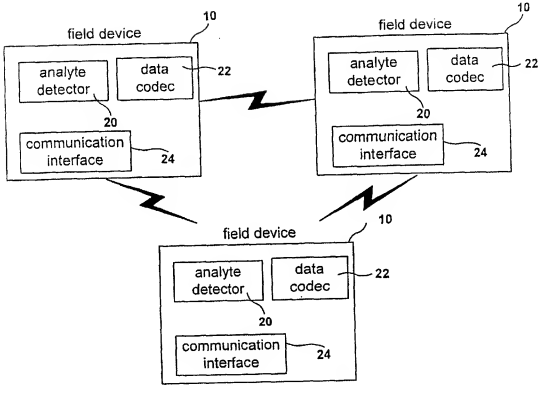

16
analyte
20
field device
10
analyte detector
data codec
22
34
user input
analyte analyzer
data storage
26
communication interface
24
32
docking device
30
computer network
18
processor
24
12
14
data codec
communication interface
Data
22

Fig. 2


field device
10
analyte detector
data codec
22
20
communication interface
24
field device
10
analyte detector
data codec
22
20
communication interface
24
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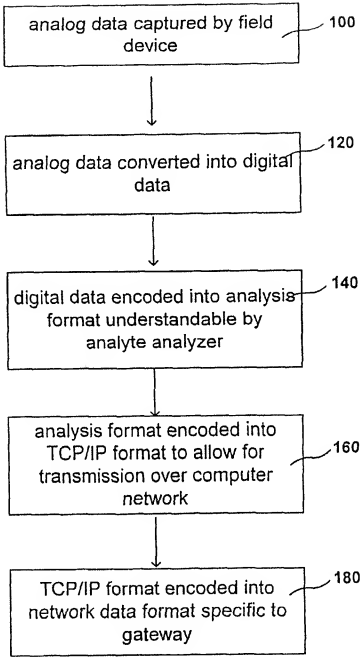
10
analyte detector
data codec
22
20
communication interface
24

Fig. 3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100
analog data converted into digital data
120
digital data encoded into analysis format understandable by analyte analyzer
140
analysis format encoded into TCP/IP format to allow for transmission over computer network
160
TCP/IP format encoded into network data format specific to gateway
180

Fig. 4

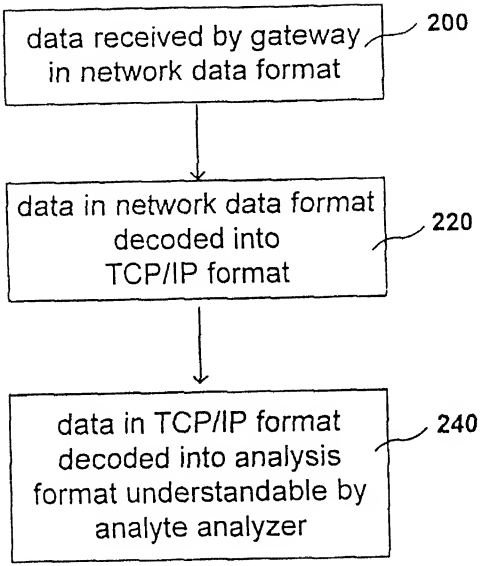
data received by gateway in network data format
200
data in network data format decoded into TCP/IP format
220
data in TCP/IP format decoded into analysis format understandable by analyte analyzer
240

Fig. 5

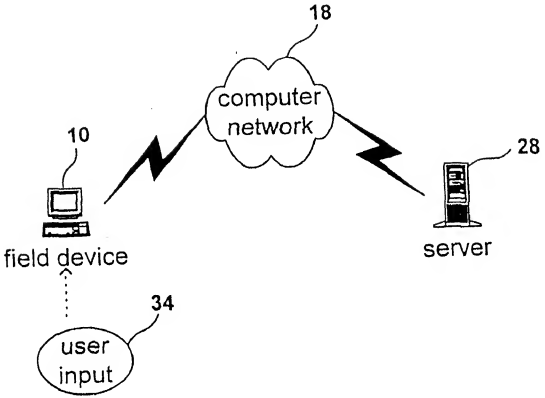

Fig. 6

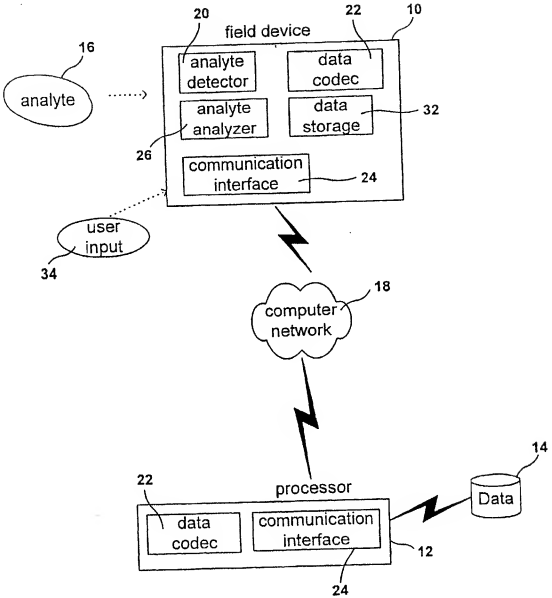
20
22
10
field device
16
analyte
analyte detector
data codec
analyte analyzer
data storage
32
26
communication interface
24
user input
34
computer network
18
14
22
processor
Data
data codec
communication interface
12
24

Fig. 7

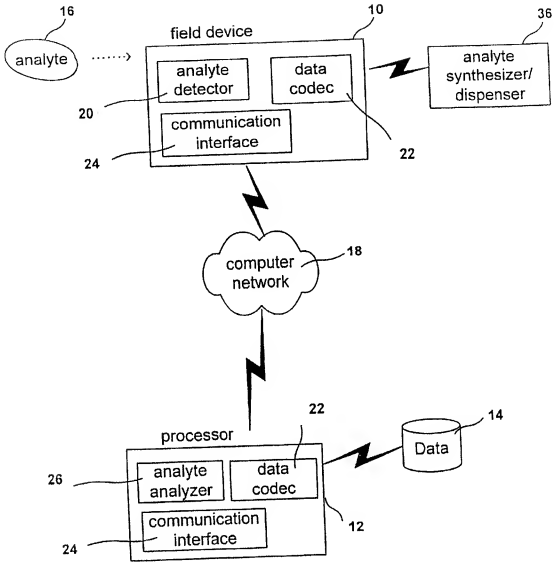
16
analyte
field device
10
analyte detector
data codec
20
communication interface
24
22
36
analyte synthesizer/ dispenser
computer network
18
22
processor
14
Data
26
analyte analyzer
data codec
12
communication interface
24

FIG. 8

COMPUTER CODE FOR PORTABLE SENSING

CROSS-REFERENCES TO RELATED APPLICATIONS

This application claims the benefit of priority from U.S. Provisional Patent Application Serial No. 60/164,022, filed on Nov. 4, 1999, U.S. Provisional Patent Application Serial No. 60/162,683, filed on Nov. 1, 1999, and U.S. Provisional Patent Application Serial No. 60/188,307, filed on Mar. 10, 2000, all of which are hereby incorporated by reference as if set forth in full in this document.

FIELD OF THE INVENTION

This invention generally relates to the detection and transmission of sensory data. More particularly, the present invention relates to computer code(s) for detecting and transmitting sensory data from one portable device to another for analytic purposes.

BACKGROUND OF THE INVENTION

Techniques and devices for detecting a wide variety of analytes in fluids such as vapors, gases and liquids are well known. Such devices generally comprise an array of sensors that in the presence of an analyte produce a unique output signature. Using pattern recognition algorithms, the output signature, such as an electrical response, can be correlated and compared to the known output signature of a particular analyte or mixture of substances. By comparing the unknown signature with the stored or known signatures, the analyte can be detected, identified and quantified. Examples of such detection devices can be found in U.S. Pat. No. 5,571,401(by Lewis et al. and assigned to California Institute of Technology); U.S. Pat. No. 5,675,070 (by Gelperin and assigned to NCR Corporation); U.S. Pat. No. 5,697,326 (by Mottram et al. and assigned to British Technology Group Limited); U.S. Pat. No. 5,788,833 (by Lewis et al. and assigned to California Institute of Technology); U.S. Pat. No. 5,807,701 (by Payne et al. and assigned to Aromascan PLC); and U.S. Pat. No. 5,891,398 (by Lewis et al. and assigned to California Institute of Technology), the disclosures of which are incorporated herein by reference.

Concurrent with the development of better detection techniques for detecting analytes, there is an emerging need to develop methods and devices to efficiently transmit the collected sensory data for swift analysis. Under some prior customary practices, the sensory data were first captured and then physically transported back to a laboratory or some other designated facility for subsequent analysis. Very often, analyses on these data would not be performed until a substantial period of time had elapsed and consequently their results would not be available for hours, days or even weeks.

Timely transmission and analysis of sensory data for detected analytes have tremendous applications in a variety of areas. There are many instances where it is desirable to obtain results on the analysis of the sensory data in a timely manner. For example, in a hospital/medical environment, it would be greatly beneficial if data collected from a patient can be transmitted quickly to a laboratory to determine the cause of the patient's ailments thereby allowing the doctors to prescribe the necessary treatment without any undue delay. In a similar example, medical and other related data from home monitoring devices can be collected and transmitted swiftly to the appropriate hospitals and/or authorities

to allow them to provide better response to home emergencies. In another example, in environments where the presence of certain substances can potentially lead to dangerous conditions, such as a gas leak in a foundry or a home, the swift transmission of sensory data for analysis can very well preempt an impending disaster. Clearly, there are many other situations which one could think of where the efficient transmission of sensory data will generate tremendous benefits. Hence, it would be desirable and beneficial to provide a system that is capable of timely transmitting sensory data for analysis.

In addition to the need to have timely transmission of sensory data, there is a need to provide easy access to the collective data compiled for the known analytes. The results of any detection analysis are only as good as the data which are available for comparison. At the present time, various analytes have been identified and data therefor have been compiled and stored all over the world. Perhaps, due to the voluminous amount of data that are available, these data are generally not centralized in any one particular repository but are instead separately stored at different facilities. The segregation of these data, therefore, renders a complete and accurate analysis more difficult. Hence, it would be desirable to have a system that is capable of providing better access to various data repositories thereby allowing more accurate analyses to be performed. The present invention remedies these shortcomings by providing a system of transmitting, storing and retrieving sensory information.

SUMMARY OF THE INVENTION

The present invention generally relates to detecting and transmitting analyte data from a field device to a processor. In an exemplary embodiment, the present invention provides computer codes for capturing and transmitting analyte data over a computer network such as an internet, the Internet, a local area network, a wide area network or any combination thereof.

In a specific embodiment, the present invention provides a system including computer code for capturing and transmitting analyte data pertaining to an unknown analyte. The computer code is embedded in memory, which can be at a single location or multiple locations in a distributed manner. The system has a first code directed to capturing data for the unknown analyte using a field device at a first geographic location. The system also includes a second code directed to transmitting the captured analyte data to a processor at a second geographic location via a computer network. In a preferred embodiment, the captured analyte data are transferred via a worldwide network of computers such as an internet, the Internet, a combination thereof, and the like.

In one aspect, before the captured analyte data are transmitted, the system includes computer code directed to encoding the captured analyte data by the field device into a transmissible format. The system also includes computer code directed to decoding the encoded analyte data by the processor to permit analysis to be performed. In order to analyze the captured analyte data, the system further includes computer code directed to retrieving data of known analytes from an electronic library and performing the analysis using such data. In addition, the system includes computer code directed to updating the electronic library with the captured analyte data. This code and others can be used with the present invention to perform the functionality described herein as well as others.

By transmitting the captured analyte data via a computer network, the present invention provides a system including

computer codes that is capable of transmitting analyte data in a timely and efficient manner. Consequently, analyses can be performed swiftly and results can be obtained on a more expedited basis.

Numerous benefits are achieved by way of the present invention over conventional techniques. For example, the present invention allows for the efficient transfer of analyte data from one geographic location to another geographic location thereby providing utility and applications in various areas such as hospital/medical applications, fire safety monitoring, environmental toxicology, remediation, biomedicine, material quality control, food monitoring, agricultural monitoring, heavy industrial manufacturing, ambient air monitoring, worker protection, emissions control, product quality testing, oil/gas petrochemical applications, combustible gas detection, H_2S monitoring, hazardous leak detection, emergency response and law enforcement applications, explosives detection, utility and power applications, food/beverage/agriculture applications, freshness detection, fruit ripening control, fermentation process monitoring and control, flavor composition and identification, product quality and identification, refrigerant and fumigant detection, cosmetic/perfume applications, fragrance formulation, chemical/plastics/pharmaceuticals applications, fugitive emission identification, solvent recovery effectiveness, anesthesia and sterilization gas detection, infectious disease detection, breath analysis and body fluids analysis. Additionally, the present invention uses conventional computer hardware and/or software, which make it easy to implement.

Using a distributed computer network for collecting analyte data and then performing the analysis and interpretation remotely has a number of advantages. For example, every new piece of data can be added to the electronic library thereby continually expanding the repository of knowledge. This approach allows historical data to be kept and retrieved for subsequent use. In addition, with the use of an electronic library, data can be easily shared at different physical locations thereby facilitating objective data comparison. For instance, data relating to a product can be captured at various shipment checkpoints to provide quality control on the product. Finally, by providing the capability to have a number of field devices transmit data to a central location, a large area can be monitored for safety or other purposes.

Reference to the remaining portions of the specification, including the drawings and claims, will realize other features and advantages of the present invention. Further features and advantages of the present invention, as well as the structure and operation of various embodiments of the present invention, are described in detail below with respect to accompanying drawings. In the drawings, like reference numbers indicate identical or functionally similar elements.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. **1** is a simplified schematic block diagram showing a system according to one embodiment of the present invention;

FIG. **2** is a simplified schematic block diagram showing a system according to a second embodiment of the present invention;

FIG. **3** is a simplified schematic block diagram showing a system according to a third embodiment of the present invention;

FIG. **4** is a simplified flow diagram showing the process of encoding the data in accordance with the present invention;

FIG. **5** is a simplified flow diagram showing the process of decoding the data in accordance with the present invention;

FIG. **6** is a simplified schematic block diagram showing a system according to a fourth embodiment of the present invention;

FIG. **7** is a simplified schematic block diagram showing a system according to a fifth embodiment of the present invention; and

FIG. **8** is a simplified schematic block diagram showing a system according to a sixth embodiment of the present invention.

DETAILED DESCRIPTION OF THE INVENTION AND SPECIFIC EMBODIMENTS

FIG. **1** is a simplified schematic block diagram showing a detection and transmission system **2** according to an exemplary embodiment of the present invention. This diagram is merely an example which should not limit the scope of the claims herein. One of ordinary skill in the art would recognize many other variations, modifications, and alternatives. As shown, the system **2** preferably includes a field device **10**, a processor **12** and an electronic library **14**.

The field device **10** is capable of detecting an analyte **16** and transmitting the data relating to such analyte via a computer network **18** to the processor **12** for analysis. It should be understood that the field device **10** is generally capable of communicating with other devices connected to the computer network **18**. In one embodiment, the field device **10** includes an analyte detector **20** and a data coder/decoder (codec) **22**.

The analyte detector **20** is a transducer, such as an electronic nose, capable of detecting the presence of an analyte **16** and then generating certain sensory data corresponding to a unique output signature specific to the detected analyte. The analyte detector **20** may utilize one of many different detection techniques, such as electronic nose technology, gas chromatography, and mass spectrometry etc., to detect the presence of an analyte depending on the attendant circumstances. An illustrative implementation of the analyte detector is disclosed in U.S. patent application Ser. No. 271,873, which is now U.S. Pat. No. 6,085,576, commonly assigned, and hereby incorporated by reference for all purposes.

The main function of the data codec **22** is to encode and decode data exchanged between the field device **10** and the outside world. For example, the data codec **22** receives data from the analyte detector **20** and, after appropriate encoding or formatting, relays them to the processor **12** via the computer network **18**. In other instances, data coming from the processor **12** are decoded by the data codec **22** to allow the data to be used by the field device **10**. The data encoding or formatting steps will be described in further details below.

The data communications between the field device **10** and the outside world, such as the processor **12**, can be either one-way or two-way communication. The field device **10** can act solely as a transmitter capable of only sending data to the processor **12**, or alternatively, the field device **10** can act as a transceiver capable of both sending and receiving data from the processor **12**.

The analyte detector **20** and the data codec **22** are preferably located within the same housing. The field device **10** can be a portable, handheld device such as the Palm® devices manufactured by 3Com and the Visor® produced by Handspring. By incorporating the analyte detector **20** and

the data codec **22** in a portable, handheld device, a user has the additional ability to operate in a mobile manner. This mobility is obviously greatly desirable as the need to detect the presence of analytes often arises in limiting environments where cable, phone or other pre-installed communication outlets are not readily available or accessible. In one embodiment, the analyte detector **20** is described in U.S. patent application Ser. No. 271,873, which is now U.S. Pat. No. 6,085,576, commonly assigned, and hereby incorporated by reference for all purposes. As described therein, the analyte detector **20** is integrated into a hand-held device thereby permitting a user to conduct the analyte detection in a mobile manner.

In an alternative embodiment (not shown), the data codec **22** can be located on a gateway, such as a computer, connected to the computer network **18**. Under this configuration, the field device **10** sends the captured analyte data to the gateway and the data codec **22** processes the data and forwards them to the processor **12** via the computer network **18**.

The processor **12** includes a data codec **22** and an analyte analyzer **26**. Similar to the data codec **22** in the field device **10**, the function of the data codec **22** in the processor **12** is to encode and decode data exchanged between the processor **12** and the outside world. For example, the data codec **22** receives data from the field device **10** via the computer network **18** and processes or decodes the data into a format which can be understood by the analyte analyzer **26**; similarly, the data codec **22** can also format or encode data so as to allow the processor **12** to transmit them to the field device **10**. In other instances, the data codec **22** also encodes or decodes the data so as to allow such data to be exchanged between the processor **12** and the electronic database **14**.

The analyte analyzer **26** is capable of performing analysis on a detected analyte. Using data stored in the electronic library **14** and after appropriate formatting by the data codec **22**, the analyte analyzer **26** compares the data received from the field device **10** with data retrieved from the electronic database **14** to identify the identity of the detected analyte. The results of the analysis can then be formatted by the data codec **22** for posting onto the electronic library **14**. In addition, the results can be made available to the field device **10** in a number of ways. For example, the processor **12** can directly send the results back to the field device **10** via the computer network **18**, or, the results can be formatted in HTML and displayed on a web page which can then be accessed by the field device **10** to retrieve the results.

The analyte analyzer **26** uses a number of pattern recognition algorithms to compare the output signature of the detected unknown analyte to the signatures of known analytes. Many of the algorithms are neural network based algorithms. A neural network has an input layer, processing layers and an output layer. The information in a neural network is distributed throughout the processing layers. The processing layers are made up of nodes that simulate the neurons by its interconnection to their nodes.

In operation, when a neural network is combined with a sensor array, the sensor data is propagated through the networks. In this way, a series of vector matrix multiplications are performed and unknown analytes can be readily identified and determined. The neural network is trained by correcting the false or undesired outputs from a given input. Similar to statistical analysis revealing underlying patterns in a collection of data, neural networks locate consistent patterns in a collection of data, based on predetermined criteria.

Suitable pattern recognition algorithms include, but are not limited to, principal component analysis (PCA), Fisher linear discriminant analysis (FLDA), soft independent modeling of class analogy (SIMCA), K-nearest neighbors (KNN), neural networks, genetic algorithms, fuzzy logic, and other pattern recognition algorithms. In a preferred embodiment, the Fisher linear discriminant analysis (FLDA) and canonical discriminant analysis (CDA) and combinations thereof are used to compare the output signature and the available data from the electronic library. The operating principles of various algorithms suitable for use in the present invention are disclosed (see, Shaffer et al., *Analytica Chimica Acta*, 384, 305–317 (1999)), the teaching of which are incorporated herein by reference.

In order to determine which pattern recognition algorithm is optimal for the analysis of a particular detected analyte, the processor **12** is trained using various sets of training data. The subject of training devices for classification or identification purposes for one or more substances capable of producing sensory information is covered by a series of patent applications, U.S. Patent Application Serial No. 60/188,589, filed on Mar. 10, 2000, U.S. Patent Application Serial No. 60/188,588, filed on Mar. 10, 2000, and U.S. Patent Application Serial No. 60/188,569, filed on Mar. 10, 2000, all commonly owned, and hereby incorporated by reference for all purposes.

With respect to the electronic library **14**, it generally contains signatures for various known analytes and other relevant information pertaining to these analytes. The electronic library **14** can be composed of a number of different databases. These databases can be located in one central repository, or alternatively, they can be dispersed among various distinct physical locations. These databases can be categorized and structured in various ways based on the needs and criteria of the database designer. For example, the data can be organized in a database using field descriptors. Field descriptors can include the sample name, type of data etc. Possible types of data include training data, identification data, or quality control data. As another example, a first database may contain data relating to various types of analytes collected using the same detection technique under a standardized set of conditions, and a second related database may contain miscellaneous information correlating to data contained in the first database; more specifically, a first database may contain aroma data for various types of wines, and a second database may contain additional information for each type of wine identified in the first database such as the vineyard, type of wine, year of bottling, etc. Alternatively, a database may contain data specific to one particular analyte with such data collected using different detection techniques. Methods used to create and organize databases are commonly known in the art, for example, relational database techniques can be used to logically connect these databases.

In one embodiment, as shown in FIG. **1**, the databases comprising the electronic library **14**, or a portion thereof, can be physically located separate from the processor **12**. These databases can reside on remote, distant servers on a local area network or the Internet. Under this arrangement, whenever any data are needed, the processor **12** needs to access the necessary database(s) via a communication channel to retrieve the requisite data for analysis. For example, the processor **12** can access and retrieve data from a remote database via a computer network such as a LAN or the Internet.

FIG. **6** illustrates another embodiment of the present invention. This diagram is merely an example which should

not limit the scope of the claims herein. One of ordinary skill in the art would recognize many other variations, modifications, and alternatives. In this embodiment, the electronic library **14** is located on the same machine as the processor **12**. For example, the processor **12** can reside on a server **28** hosting a website and the electronic library **14** can similarly reside on the same server **28**. With this arrangement, the electronic library **14** and the data contained therein are readily accessible for use by the processor **12**.

The data in the electronic library **14** can be stored in a number of different formats. For example, the data can be formatted into HTML documents which can then be made accessible on the Internet from any remote location.

Since data are constantly provided to the electronic library **14** during opcration of the present invention, the electronic library **14** may need to be updated on a periodic basis to keep the size of the electronic library **14** manageable. Various schemes can be used to update the electronic library **14**. In one scheme, the older data are discarded after some predetermined period of time. In another scheme, the older data are averaged and then compressed on a regular basis so as to make room for the more recently captured data. In yet another scheme, the more recent data are stored in the database only when such data represent an exception or deviation.

A number of different technologies can be used to implement the communications between the field device **10**, the processor **12** and the electronic library **14**. As to communications between the field device **10** and the processor **12**, such communications can be conducted via a computer network **18**. In order to provide a physical connection to the outside world for the transmission of captured analyte data, the field device **10** includes a communication interface **24** that is capable of being coupled to the computer network **18**. The communication interface **24** may include an Ethernet interface, an RS-232 interface, a parallel port, a universal serial bus (USB), an infrared data link, an optical interface, or an RF interface. The computer network **18** can be one of a variety of networks including a worldwide computer network, an internet, the Internet, a WAN, a LAN or an intranet. It should be understood that conventional access to the computer network is conducted through a gateway (not shown). A gateway is a machine, for example, a computer, that has a communication address recognizable by the computer network.

The field device **10** can communicate with the computer network **18** via the communication interface **24** using either wireless or wired technologies. Wireless technologies may include infrared, radio waves, satellite and microwaves. Wired technologies may include cables and modems.

FIG. 2 illustrates another embodiment of the present invention. This diagram is merely an example which should not limit the scope of the claims herein. One of ordinary skill in the art would recognize many other variations, modifications, and alternatives. As shown therein, the field device **10** can be detachably coupled to a docking device **30** which, in turn, is connected to a gateway on the computer network **18**.

FIG. 3 illustrates yet another embodiment of the present invention. This diagram is merely an example which should not limit the scope of the claims herein. One of ordinary skill in the art would recognize many other variations, modifications, and alternatives. As shown therein, field devices **10** may be able to communicate with one another directly. In this device-to-device type of communication, infrared signals are generally used.

As to communications between the processor 12 and the electronic library **14**, such communications can also be conducted via a computer network **18** or other communication links such as a modem. Similarly, the processor **12** also includes a communication interface **24** to allow the processor 12 to communicate with other devices. Also, as mentioned above, depending on various requirements, the electronic library **14** can reside on the same machine as the processor **12**, thereby reducing communication overhead and costs.

The field device **10** generally performs the following steps before the captured analytc data are delivered to the computer network **18** for transmission: (1) capturing analyte data in analog form; (2) converting the analog data into digital data; (3) encoding digital data into an analysis format; (4) encoding data in analysis format into a TCP/IP format; and (5) encoding data in TCP/IP format into a specific network data format.

At the receiving end, the processor 12 generally performs the following steps to decode the encoded data: (1) receiving data in specific network data format; (2) decoding the received data into TCP/IP format; and (3) decoding the data in TCP/IP format into an analysis format.

Details of these steps will now be described with reference to FIGS. **4** and **5**. FIG. **4** illustrates the various data encoding formats needed to convert the analog data from the detected analyte into a transmissible format for transmission to the processor **12**. This diagram is merely an example which should not limit the scope of the claims herein. One of ordinary skill in the art would recognize many other variations, modifications, and alternatives. At step **100**, analog data from the detected analyte are first captured by the analyte detector **20** in the field device **10**. The analyte detector **20** acting as a transducer then converts the analog data into digital signals at step **120**. At step **140**, the digital signals are encoded into an analysis format which can be understood by the analyte analyzer **26**. This format can be either proprietary or well-known. Any format can be used as long as the analyte analyzer **26** is capable of handling such format. While it is not necessary that the format used by the field device **10**, the processor **12**, and the electronic library **14** must be the same, a standardized format is preferred since format-conversion overhead can be saved. At step **160**, the formatted data are further encoded into a format which can be transmitted over the computer network **18**, such as the TCP/IP format. This step **160** is important if the formatted data are to be sent to the processor **12** via a computer network **18**, such as the Internet, which contains numerous sub-networks having different network data formats. At step **180**, the data in TCP/IP format are encoded into the specific network data format which the gateway to the computer network **18** can understand.

FIG. 5 illustrates the various data decoding formats needed to convert the transmitted data received from the field device **10** to permit analysis by the processor **12**. This diagram is merely an example which should not limit the scope of the claims herein. One of ordinary skill in the art would recognize many other variations, modifications, and alternatives. At step **200**, data transmitted from the field device **10** via the computer network **18** are received by the gateway in a network data format specific to the gateway. At step **220**, the data in the network data format are decoded into the TCP/IP format. At step **240**, the data in TCP/IP format are further decoded into an analysis format which can be used by the analyte analyzer **26** for analysis.

The present invention can be used in a number of different ways. In one mode of operation, a user first uses the field

device **10** to capture information on an unknown analyte **16**, and then relays the captured information to the processor **12** for analysis. More specifically, the analyte detector **20** is used to detect the presence of an unknown analyte **16**. The analyte detector **20** then accordingly generates a unique output signature for this unknown analyte **16**. The unique output signature is next relayed to the data codec **22** and encoded for transmission to the processor **12**.

The data codec **22** in the processor **12** accepts the output signature from the field device **10** and then, after appropriate processing, passes it onto the analyte analyzer **26** for analysis. Depending on the detection technique used to detect the unknown analyte **16** and other relevant information which can be provided by the user, the processor **12** accesses the electronic library **14** retrieving the pertinent data corresponding to the signatures of various known analytes. The analyte analyzer **26** then compares the output signature with these known signatures to ascertain the identity of the detected analyte. If desired, the results of the comparison are transmitted to the field device **10** from the processor **12** for use by the user. Alternatively, the results of the comparison can be posted onto a web page for retrieval by the field device **10**.

Optionally, if the output signature of the detected analyte is determined to be not currently included in the electronic library **14**, the processor **12** can then appropriately update the electronic library **14** to reflect the new output signature and its accompanying information.

For example, the present invention can be used to detect chemical leaks. Data relating to the harmful chemical are captured by the field device **10** and relayed to the processor **12**. The processor **12** compares the captured data to data available from the electronic library **14** to ascertain the identity of the chemical. Results of the comparison are then sent to the field device **10** to enable the user to initiate the necessary remedial measures, if any, to limit further damage. Optionally, in the event that the identity of the chemical cannot be determined using the data currently existing in the electronic library **14**, the processor **12** will update the electronic library **14** to reflect the discovery of this "new" chemical for future identification.

In another mode of operation, field devices **10** are capable of communicating and exchanging data with one another using their respective communication interfaces **24**. The primary purpose here is to allow sharing of data between the two devices **10**. In the event that multiple field devices **10** (employing the same detection technique) are used to detect the same unknown analyte, data collected from these devices **10** can be used by the processor **12** for calibration purposes to provide for any use-to-use variability of a field device **10**.

In another embodiment, as shown in FIG. **6**, the field device **10** can be a remote computer capable of connecting to the Internet, the processor **12** can be an interactive website residing on a remote server **28** connected to the Internet, and the electronic library **14** can be located on the same remote server **28**. In a mode of operation in accordance with this embodiment, the user uses the field device **10** to retrieve information for certain analytes which are similar or related to a known, desired analyte. More specifically, the user enters the relevant information for the desired analyte into the field device **10**. The field device **10**, via the communication interface **24**, transmits the entered information to the processor **12**. The processor **12** processes the entered information and retrieves from the electronic library **14** the corresponding signature for the desired analyte.

Having retrieved the corresponding signature, the processor **12** then searches the electronic library **14** to identify a group of analytes which are similar or related to the analyte desired by the user by comparing the corresponding signature with other known signatures.

Additional information concerning each analyte within this identified group can be retrieved from other databases, if necessary. The identity of each analyte within the identified group and all the accompanying information are subsequently transmitted to the field device **10** for use by the user. Optionally, other information entered by the user can be used to narrow the identified group of analytes.

For example, the present invention can be used in a wine store to help consumers identify a wine list based on their personal tastes and preferences. In addition to different tastes, most wines also have their own distinctive aromas. Therefore, an electronic library storing data on wine aromas and other relevant information can be created. If a consumer has previously enjoyed a particular wine and can provide sufficient information about that wine, the present invention can be used to compile a list of comparable wines which the consumer may similarly enjoy. Using the present invention, this wine list can be further narrowed based on other factors such as country of origin, price, availability, shipping costs, and prior selections, etc.

FIG. **7** illustrates an alternate embodiment of the present invention. This diagram is merely an example which should not limit the scope of the claims herein. One of ordinary skill in the art would recognize many other variations, modifications, and alternatives. As shown therein, certain components of the processor **12**, such as the analyte analyzer **26**, can reside within the field device **10**. The analyte analyzer **26** is included within the field device **10** as opposed to the processor **12** and the field device **10** further includes a data storage area **32**. With this particular configuration, the present invention may be operated in the following manner. The user enters a request **34** into the field device **10** for data relating to certain specified, known analytes. The field device **10** then transmits the request **34** to the processor **12**. The processor **12**, in turn, retrieves the relevant data from the electronic library **14** in accordance with the request **34** and forwards the requested data to the field device **10**. Upon receipt of the requested data, the field device **10** stores them in a data storage area **32** for subsequent use. When the field device **10** is used to detect an unknown analyte **16**, data in the data storage area **32** are readily available for use by the analyte analyzer **26** to compare and identify the detected analyte **16**.

By having the analyte analyzer **26** and the data storage area **34** incorporated into the field device **10**, the time required for analysis can be shortened. For example, prior to entering a particular area, if the user knows that there is a relatively high probability of presence of certain known analytes in that area, the user can download the signatures of these known analytes onto the field device **10** ahead of time. With the signatures readily available within the field device **10**, the output signature of the detected analyte **16** can be compared against these known signatures first. Therefore, there may not be a need to connect to the processor **12** thereby allowing the analysis to be performed more quickly. Connection to the processor **12** only needs to be made when none of the downloaded signatures matches with that of the detected analyte **16**.

The present invention can be used in many different applications. In certain embodiments, the system of the present invention can be used for monitoring medical con-

ditions and disease processes. For instance, WO 98/29563, published Jul. 9, 1998, and incorporated herein by reference, discloses a method for monitoring conditions in a patient wherein a sample is obtained from a patient over a period of time. The samples are then flowed over a gas sensor and a response is measured. Thereafter, the response is correlated with known responses for known conditions. The conditions include, but are not limited to, the progression and/or regression of a disease state, bacterial infections, viral, fungal or parasitic infections, the effectiveness of a course of treatment and the progress of a healing process.

In certain instances, the patient is in a nursing home, primary residence or hospital. The patient uses the field device **10** to capture data on an analyte such as, but not limited to, a breath sample, which the patient provides. The data on the breath sample can be optionally transmitted over the Internet or intranet to the processor **12** and then be subsequently analyzed or read by a medical professional at a health company, doctors office or hospital. Using the system of the present invention, real time home health management is realized.

In certain aspects, the analyte data, such as olfaction data, vital signs and any other symptoms of the patient are transmitted to a second location. The data can then be analyzed and the medical condition and disease process monitored. Thereafter, the patient can access the diagnostic information on a private Web site for further instructions and treatment.

In other aspects, the system of the present invention can be used for monitoring chronic diseases which generally have associated with them distinctive odors or smells. For example, the system of the present invention can be used for monitoring medical conditions in a respiring subject. For instance, WO 98/39470, published Sep. 11, 1998, and incorporated herein by reference, discloses a method for detecting the occurrence of a condition in a respiring subject. The method comprises introducing emitted respiratory gases to a gas sensing device, detecting certain species present in the gas and correlating the presence of the species with certain conditions.

A wide variety of conditions can be ascertained using this aspect of the present invention. These conditions include, but are not limited to, halitosis, ketosis, yeast infections, gastrointestinal infections, diabetes, alcohol, phenylketonuria, pneumonia, and lung infections. Those of skill in the art will know of other conditions and diseases amenable to the method and system of the present invention.

In yet another embodiment, the system of the present invention can be used for monitoring conditions and disease processes in female patients. For instance, WO 99/09407, published Feb. 25, 1999, and incorporated herein by reference, discloses a method for detecting the occurrence of a condition in a female patient comprising obtaining a sample of gaseous or volatile substance from the vaginal region of the patient, detecting the gas and correlating the detection with the occurrence of a condition. A wide variety of conditions can be ascertained using this aspect of the present invention. These conditions include, but are not limited to, cervical cancer, ovarian or uterine cancer, HIV, sexually transmitted diseases, cytomegalovirus, yeast infections, pregnancy and Chlamydia.

In still yet another embodiment, the system of the present invention can be used for monitoring conditions and disease processes using a device that affixes to a portion of the skin on a subject. For instance, WO 99/09408, published Feb. 25, 1999, and incorporated herein by reference, discloses a method for detecting a condition of a subject with a device that is adapted to be affixed to the subject and having a gas sensing means disposed so as to detect gases and volatile species emanating from a portion of the skin. A wide variety of conditions can be ascertained using this aspect of the present invention. These conditions include, but are not limited to, skin cancer, diabetes, heart disease, heavy metal in the subject and drugs.

FIG. **8** illustrates yet another embodiment of the present embodiment. This diagram is merely an example which should not limit the scope of the claims herein. One of ordinary skill in the art would recognize many other variations, modifications, and alternatives. As shown therein, the present invention includes an analyte synthesizer or dispenser **36**. The analyte synthesizer **36** is a device which is capable of synthesizing or dispensing analyies based on input information and parameters. The analyte synthesizer/dispenser **36** can be coupled to the field device **10** to receive the relevant analyte information. A conventional analyte synthesizer is the "iSmell™" synthesizer, or personal scent synthesizer available from Digiscents (Oakland California). The iSmell™ synthesizer is a software-controlled computer peripheral device that is capable of emitting a broad range of fragrances, smells and aromas using a combination and synthesis of primary odorants.

This embodiment may be used in the following manner. The signature of a known analyte is relayed by the field device **10** to the analyte synthesizer/dispenser **36** and thereafter the known analyte is reconstructed to produce either the actual fragrance, aroma, scent, or smell or a simulated version thereof. In addition, other analytes which are similar to the known analyte can also be reconstructed to offer a wider range of selection.

This embodiment including the analyte synthesizer/dispenser **36** can be used for various purposes. For example, an electronic library **14** can contain signatures of a myriad of consumer products including, but not limited to, perfumes, cigars, liquor, coffee, cosmetics, lipsticks, tobacco and wine. Using the system of the present invention, a consumer can, for example, physically smell a reconstructed sample of a particular brand of perfume having a characteristic signature, and if the consumer enjoys this brand of perfume, it is possible to suggest and then synthesize other perfumes with similar signatures that the consumer may also enjoy to provide a wider consumer choice.

The present invention can further be used for medical purposes, for example, delivering an odorant for inhalation via a computer network so as to stimulate the male sexual response. As described in U.S. Pat. No. 5,885,614, which issued to Hirsch, on Mar. 23, 1999, the use of odorants are useful for inducing or enhancing an erection, and as aids for a non-invasive treatment of male vasculogenic impotence. As described therein, the administration of odorants for inhalation by a male individual having a normal olfactory ability effectively increased penile blood flow from about 2–40%, and enhanced sexual arousal. Preferred odorants are those that provided a 20–40% increase in blood flow to the penis, which includes lavender, oriental spice, cola and orange, and odorant mixtures of lavender and pumpkin pie, doughnut and black licorice, and pumpkin pie and doughnut. The odorants are useful as adjuvants to augment penile blood flow and as aids in the treatment of male impotence, and to enhance sexual arousal in normal males, i.e., those without sexual dysfunction. The signature of the desired odorant is transmitted via the Internet to the analyte synthesizer/dispenser **36**. The desired odorant is thereafter synthesized and/or dispensed to the male by inhalation.

It is understood that, based on the disclosure provided herein, the system of the present invention, or portions thereof, and the functionality described in connection therewith, can be implemented in many different ways by one of ordinary skill in the art. In an exemplary embodiment, the system of the present invention and its functionality are implemented using computer code and/or software programming techniques in a modular manner. However, many other ways of implementing the present invention are available as should be apparent to a person of ordinary skill in the art.

It is also understood that the examples and embodiments described herein are for illustrative purposes only and that various modifications or changes in light thereof will be suggested to persons skilled in the art and are to be included within the spirit and purview of this application and scope of the appended claims. All publications, patents, and patent applications cited herein are hereby incorporated by reference for all purposes in their entirety.

What is claimed is:

1. A system comprising memory including a computer code product for detecting and transferring data pertaining to an analyte from a first device to a second device, said memory comprising:

a code directed to capturing analyte data pertaining to said analyte using said first device;

a code directed to transmitting said analyte data via a computer network to said second device;

a code directed to encoding said analyte data into a transmissible format for transmission via said computer network to said second device;

a code directed to decoding said analyte data in said transmissible format; and

a code directed to directing said second device to perform an analysis on said analyte data.

2. A system according to claim **1**, wherein said code directed to encoding further comprises:

a code directed to capturing said analyte data in an analog format;

a code directed to converting said analyte data in analog format into a digital format;

a code directed to encoding said analyte data in digital format into an analysis format;

a code directed to encoding said analyte data in analysis format into TCP/IP format; and

a code directed to encoding said analyte data in TCP/IP format into a network-specific data format.

3. A system according to claim **1**, wherein said code directed to decoding further comprises:

a code directed to decoding said analyte data in a network-specific data format into TCP/IP format; and

a code directed to decoding said analyte data in TCP/IP format into an analysis format.

4. A system according to claim **1**, further comprising a code directed to retrieving signatures of known analytes from an electronic library.

5. A system according to **4**, further comprising a code directed to analyzing said analyte data using said retrieved signatures.

6. A system according to claim **5**, wherein said codes directed to retrieving and analyzing respectively are executed by said second device.

7. A system according to claim **4**, wherein said electronic library includes one or more databases.

8. A system according to claim **5**, further comprising a code directed to displaying result of execution of said code directed to analyzing on a web page.

9. A system according to claim **4**, further comprising a code directed to updating said electronic library with said analyte data;

wherein said code directed to updating is executed by said second device.

10. A system according to claim **1**, wherein said transmission of said analyte data is conducted via wireless communications.

11. A system according to claim **10**, wherein said wireless communications are implemented using communications technologies selected from a member of a group consisting of infrared technology, satellite technology, microwave technology and radio wave technology.

12. A system according to claim **1**, wherein said transmission of said analyte data is conducted via wired communications.

13. A system according to claim **1**, wherein said computer network is selected from a member of a group consisting of a worldwide computer network, an internet, the Internet, a wide area network, a local area network, and an intranet.

14. A system according to claim **1**, wherein said first device is a handheld device.

15. A system according to claim **1**, wherein said analyte data is olfaction data.

16. A system according to claim **1**, wherein said system is used in an application selected from a group consisting of hospital/medical applications, fire safety monitoring, environmental toxicology, remediation, biomedicine, material quality control, food monitoring, agricultural monitoring, heavy industrial manufacturing, ambient air monitoring, worker protection, emissions control, product quality testing, oil/gas petrochemical applications, combustible gas detection, H_2S monitoring, hazardous leak detection, emergency response and law enforcement applications, explosives detection, utility and power applications, food/beverage/agriculture applications, freshness detection, fruit ripening control, fermentation process monitoring and control, flavor composition and identification, product quality and identification, refrigerant and fumigant detection, cosmetic/perfume applications, fragrance formulation, chemical/plastics/pharmaceuticals applications, fugitive emission identification, solvent recovery effectiveness, anesthesia and sterilization gas detection, infectious disease detection, breath analysis and body fluids analysis.

17. A system including memory and computer codes for detecting and transferring analyte data, said system comprising:

a code directed to capturing said analyte data using a first device or a second device;

a code directed to converting said analyte data into a transmissible format;

a code directed to transmitting said converted analyte data in said transmissible format from said first device or said second device; and

a code directed to receiving said transmitted data in said transmissible format using said first device or said second device;

wherein said first device and said second device are functionally equivalent.

18. A system according to claim **17**, wherein said transmission of said converted analyte data is conducted via wireless communications.

19. A system according to claim **17**, wherein said wireless communications are implemented using infrared technology.

20. A system including memory and computer codes for identifying an analyte, said system comprising:

a code for capturing at a first location data pertaining to said analyte whose identity is unknown;

a code for transmitting said data from said first location via a computer network;

a code for receiving said data at a second location; and

a code for comparing said received data at said second location to data pertaining to known analytes, thereby identifying said analyte.

21. A system according to claim **20**, further comprising a code for retrieving said data pertaining to known analytes from an electronic library.

22. A system according to claim **20**, further comprising a code directed to updating said electronic library with said received data.

23. A system according to claim **20**, further comprising a code directed to making result of execution of said code for comparing available at first location.

24. A system including memory and computer codes for analyzing data pertaining to a detected analyte, said system comprising:

a code directed to transmitting data pertaining to known analytes from an electronic library to a first location via a computer network;

a code directed to receiving said data pertaining to known analytes at said first location; and

a code directed to analyzing said data pertaining to said detected analyte using said received data pertaining to known analytes at said first location and generating an analysis result.

25. A system according to claim **24**, further comprising:

a code directed to receiving input from a user; and

a code directed to using said input during execution of said code directed to analyzing.

26. A system including memory and computer codes for delivering analyte data, said system comprising:

a code directed to receiving a request from a user requesting data of a known analyte;

a code directed to retrieving data of said known analyte from an electronic library; and

a code directed to transmitting said retrieved data to said user via a computer network.

27. A system according to claim **26**, further comprising a code directed to synthesizing said known analyte using said retrieved data.

28. A system according to claim **26**, further comprising:

a code directed to retrieving data of analytes which are similar to said known analyte from said electronic library;

a code directed to transmitting said retrieved data of analytes which are similar to said known analyte to said user via said computer network; and

a code directed to synthesizing said analytes which are similar to said known analyte using said retrieved data thereof.

29. A system including memory and computer codes for delivering medicine to a patient at a remote location, said system comprising:

a code directed to identifying needs of said patient at said remote location;

a code directed to transmitting information relating to said needs to a processor via a computer network;

a code directed to receiving from said processor information in response to said needs; and

a code directed to using information received from said processor to synthesize or dispense said medicine to satisfy said needs of said patient.

30. A system according to claim **29**, wherein said code directed to identifying includes a code directed to capturing information relating to said needs using a field device.

31. A system including memory and computer codes for facilitating consumer choice, said system comprising:

a code directed to facilitating selection of a first consumer product having a known signature; and

a code directed to comparing said known signature with a plurality of signatures so as to allow a similar signature indicative of a second consumer product to be selected.

32. A system according to claim **31**, wherein said code directed to comparing includes:

a code directed to retrieving data pertaining to said plurality of signatures from an electronic library; and

a code directed to synthesizing each of said plurality of signatures using said retrieved data so as to facilitate selection of said similar signature.

33. A system according to claim **32**, wherein said code directed to selecting includes:

a code directed to retrieving data pertaining to said known signature from said electronic library; and

a code directed to synthesizing said known signature using said retrieved data pertaining to said known signature.

* * * * *

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[54] PROCESS TO DEODORIZE AN ODOROUS POLY(MONO-1-OLEFIN)

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[58] Field of Search 528/480, 483, 490, 491, 528/494, 496, 498, 499

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[57] ABSTRACT

A process is provided comprising contacting under deodorizing conditions, at least one deodorizing composition with at least one poly(mono-1-olefin), wherein said deodorizing conditions comprise, a standard pressure from about 1 to about 5, and a standard temperature from about 0.5 to about 2.5.

33 Claims, No Drawings

PROCESS TO DEODORIZE AN ODOROUS POLY(MONO-1-OLEFIN)

BACKGROUND OF THE INVENTION

This invention relates to a process to deodorize an odorous poly(mono-1-olefin).

Poly(mono-1-olefin)s have been used in a wide variety of applications. For example, poly(mono-1-olefin)s have been fabricated into molded articles such as pipes, films, fibers, and containers. However, one persistent problem that has limited the development of poly(-mono-1-olefin) applications has been the generation of odorous compounds during the production of the poly(-mono-1-olefin). These odorous compounds are then incorporated into the poly(mono-1-olefin) material. After incorporation, these odorous compounds interfere with the utilization of the poly(mono-1-olefin) material in applications where such odors would be undesirable. An example of this occurs when a poly(mono-1-olefin) material, such as polypropylene, is used to fabricate a container, for a liquid or a solid product that is meant for human consumption, and that poly(mono-1-olefin) container imparts a distasteful olfactory impact upon the consumer of the product.

Considering the subjectiveness of determining an odor's quality, it is best if there are not any odorous compounds in the poly(mono-1-olefin) material at all. However, it is hard, if not impossible sometimes, to determine exactly which compound, in a group of compounds, is odorous. Given the difficulties in determining which compounds are odorous, it is reasonable to eliminate as many suspected odorous compounds as possible from the poly(mono-1-olefin). However, current methods in the art to eliminate odors from poly(mono-1-olefin) materials have not kept pace with consumer demand. Furthermore, most solutions to this odor problem involve such materials and conditions as oxidizing agents and/or high pressure (>100 MPa) equipment. One reason for this lack of development has been due, in part, to the aliphatic, close-packed, molecular structure of these poly(mono-1-olefins). This invention provides an alternative method of odor elimination/reduction.

SUMMARY OF THE INVENTION

It is an object of this invention to provide a process to deodorize an odorous solid poly(mono-1-olefin).

It is another object of this invention to provide a process to lower the odor intensity score (as defined herein) of an odorous solid poly(mono-1-olefin).

It is still another object of this invention to provide a process to neutralize the odor quality score (as defined herein) of an odorous solid poly(mono-1-olefin).

It is still yet another object of this invention to provide a process to lower the odor impact (as defined herein) score of an odorous solid poly(mono-1-olefin).

In accordance with this invention, a process is provided comprising contacting under deodorizing conditions, at least one deodorizing composition with at least one poly(mono-1-olefin) that is in the solid state, wherein said deodorizing conditions comprise, a standard pressure (as defined herein) from about 1 to about 5 and a standard temperature (as defined herein) from about 0.5 to about 2.5.

In accordance with another embodiment of this invention a process is provided consisting essentially of contacting under deodorizing conditions, at least one deodorizing composition with at least one poly(mono-1-olefin), wherein said deodorizing conditions consist essentially of, a standard pressure from about 1 to about 5 and a standard temperature from about 0.5 to about 2.5.

In accordance with yet another embodiment of this invention a process is provided consisting of contacting under deodorizing conditions, at least one deodorizing composition with at least one poly(mono-1-olefin), wherein said deodorizing conditions consist of, a standard pressure from about 1 to about 5 and a standard temperature from about 0.5 to about 2.5.

The invention as disclosed herein may suitably be practiced in the absence of any steps, parameters, process conditions, or components not specifically disclosed herein.

One of the advantages of this invention is that the solid poly(mono-1-olefin) composition can remain in the solid state during this process thereby lowering production costs.

DETAILED DESCRIPTION OF THE INVENTION

Poly(mono-1-olefin)s

The term poly(mono-1-olefin) is defined for the purposes of this application to mean both poly(mono-1-olefins) and poly(mono-1-olefin). In the first case more than one type of mono-1-olefin can be present in the molecular structure. In the second case only one type of mono-1-olefin can be present in the molecular structure. By "type" the applicants mean the molecular arrangement and composition of the mono-1-olefin monomer. Generally, these terms are also referred to as copolymers and homopolymers, respectively.

In general, this invention is broadly applicable to any poly(mono-1-olefin). These poly(mono-1-olefin)s can have an atactic, syndiotactic, or isotactic molecular structure. Furthermore, such a poly(mono-1-olefin) can have a mixture of these types of molecular structures in its macrostructure.

If the poly(mono-1-olefin) is a copolymer it can have a random or regular molecular olefin structure. That is, a random copolymer would have at least two different mono-1-olefins arranged in a random order in the molecular chain. A regular copolymer could either have an alternating or block molecular olefin structure. An alternating molecular olefin structure would have at least two different mono-1-olefins arranged in a repeating order in the molecular chain. For example, if four mono-1-olefins A, B, C, and D were polymerized in an alternating molecular olefin structure a possible example would be ABCDABCD. A block molecular olefin structure would have at least two different mono-1-olefins arranged in a segmented repeating order in the molecular chain. For example, if four mono-1-olefins A, B, C, D were polymerized in a block molecular olefin structure a possible example would be AAABBBCCCDDD.

The molecular weight of these poly(mono-1-olefins) can be from about 1,000 to about 20,000,000 as determined by gel permeation chromatography. Preferably, the molecular weight is from about 5,000 to about 2,000,000 and most preferably from 10,000 to 1,000,000 due to ease of production, use, and economic factors. An example of a molecular weight determination method would be using a Waters 150C chromatograph operated at 140° C. with 1,2,4 trichlorobenzene as a carrier solvent for determining the molecular weight of

a polyethylene material separated by size exclusion or gel permeation chromatographic columns, SEC or GPC respectively.

Examples of poly(mono-1-olefin)s which can be used in this invention include, but are not limited to, polyethylene, polypropylene, poly(1-butene), poly(3-methyl-1-butene), poly(1-pentene), poly(3-methyl-1-pentene), poly(4-methyl-1-pentene), poly(1-hexene), poly(3-ethyl-1-hexene), as well as, mixtures of two or more of said poly(mono-1-olefin)s.

The mono-1-olefins which can be polymerized into poly(mono-1-olefin)s can be characterized by the following formula:

wherein each X is independently selected from the group consisting of hydrogen, fluorine, chlorine, bromine, and iodine; and wherein the R group is selected from the group consisting of hydrogen, fluorine, chlorine, bromine, iodine, and alkyl radicals; and wherein said alkyl radicals have either a linear or branched molecular structure; and wherein said alkyl radicals consist essentially of carbon and hydrogen; and wherein the number of carbon atoms in each alkyl radical is from 1 to about 32 inclusive, preferably from 1 to 20, and most preferably from 1 to 10. Examples of these mono-1-olefins are ethylene, vinyl chloride tetrafluoroethylene, propylene, 1-butene, 3-methyl-1-butene, 1-pentene, 3-methyl-1-pentene, 4-methyl-1-pentene, 1-hexene, 1-heptene, 1-octene, 1-nonene, 1-decene, dodecene, tetradecene, hexadecene, as well as, mixtures of two or more of said mono-1-olefins. These mono-1-olefins can be polymerized either alone or in combination with one or more of the others. Furthermore, they can be polymerized with any of the known catalyst systems or polymerization techniques.

Prior to deodorizing the odorous poly(mono-1-olefin), it can be contacted with a metal complexing compound to further enhance the use of this process. For example, any low molecular weight (less than 1,000) alcohol, dialcohol, ketone, or diketone could be used to complex residual metals in the poly(mono-1-olefin). Thereafter, during the deodorizing process a substantial portion of these metals could be removed. Examples of metal complexing compounds include, but are not limited to, methanol, ethanol, propanol, 1,3-propanediol, 1,4-butanedione, acetylacetone, as well as mixtures of two or more of said metal complexing agents.

DEODORIZING COMPOSITIONS

The deodorizing composition can be any compound in which the poly(mono-1-olefin) is insoluble at the process conditions described below. Additionally, the deodorizing composition should be able to show supercritical behavior at some temperature and pressure. That is, when the composition is compressed and heated to conditions above its critical point, the substance becomes a supercritical fluid. That is, as the critical point is approached, its isothermal compressibility tends to infinity and its density changes dramatically. Examples of deodorizing substances are ammonia, boron trifluoride, carbon dioxide, krypton, phosphine, chlorotrifluorosilane, silane, silicon tetrafluoride, xenon, monochlorodifluoromethane, trifluoromethane, monofluoromethane, dichlorodifluoromethane, monobromotrifluoromethane, monochlorotrifluoromethane, tetrafluoromethane, acetylene, ethylene, 1,1-difluoroethylene, ethane, dimethylether, propadiene, methylacetylene, propylene, propane, isobutane, perfluorobutane, methanol, ethanol, propanol, water, as well as, mixtures of two or more of said deodorizing compositions. The most preferred deodorizing substance is carbon dioxide due to ease of use and availability. Additionally, mixtures of different deodorizing substances could be used in this process at the same time.

PROCESS CONDITIONS

The most important factors to consider in performing the invention are the pressure and the temperature of the deodorizing composition upon its initial contact with the poly(mono-1-olefin).

In general, the pressure used, is from a standard pressure of about 1 to about 5.0. The standard pressure is defined as the pressure used during the invention divided by the critical pressure for that substance. Since the pressure units are equal they cancel out. The standard pressure is also known in the art as the reduced pressure. The critical pressures of deodorizing compositions can be readily determined by persons skilled in the art. Values of critical pressures for several compositions have been tabulated. For example, the 56th Edition of the Handbood of Chemistry and Physics on p. F-85 through F-86 discloses several compositions and their critical pressure. Preferably the pressure is from about a standard pressure of about 1 to about 4.0. However, most preferably the pressure is from a standard pressure of 1 to a standard pressure of 3, inclusive. An example is the use of carbon dioxide, which has a critical pressure of 1071 psia or 7.38 megapascals. In general, the pressure at which the carbon dioxide is employed is in the range of about 7.38 megapascals to about 37 megapascals. Preferably, the pressure is in the range of about 7.38 megapascals to about 30 megapascals and more preferably from 7.38 megapascals to 22 megapascals inclusive. However, for carbon dioxide the most preferred range is 8 to 16 megapascals inclusive due to ease of use and processing conditions.

In general, the temperature used is from a standard temperature of about 0.5 to a standard temperature of 2.5. The standard temperature is defined as the temperature, in Kelvins, used during the process divided by the critical temperature, in Kelvins, of the deodorizing substance. The standard temperature is also known in the art as the reduced temperature. The critical temperatures of deodorizing compositions can be readily determined by persons in the art. Values of critical temperatures for several compositions have been tabulated. For example, the 56th Edition of the Handbook of Chemistry and Physics on p. F-85 through F-86 discloses several compositions and their critical temperatures. However, the important criteria for this process variable is that the poly(mono-1-olefin) should not melt at the temperature employed, in other words the poly(mono-1-olefin) must remain substantially solid during this process. However, it is preferably that the temperature used is from a standard temperature of about 0.75 to about 1.5, and most preferably the temperature is from a standard temperature of 0.90 to a standard temperature of 1.10. An example is the use of carbon dioxide which has a critical temperature of 304 Kelvins. In general, the temperature at which carbon dioxide is

employed, is from about 150 Kelvins to about 760 Kelvins, preferably about 230 Kelvins to about 460 Kelvins, and most preferably 275 Kelvins to 335 Kelvins.

Other factors to consider are the flow rate and the deodorization time. The flow rate of the deodorizing composition pass and/or through the poly(mono-1-olefin) is from about 1 m^3/s (cubic meters per second) to about 1,000 m^3/s. Preferably from about 2 m^3/s to about 100 m^3/s and most preferably from 4 m^3/s to 50 m^3/s inclusive. The deodorizing time, or the amount of time the poly(mono-1-olefin) is subjected to the above conditions, is from about 1 minute to about 10 hours. Preferably about 5 minutes to about 5 hours and most preferably from about 10 minutes to about 120 minutes.

EXAMPLE

This example is provided to further assist a person skilled in the art with understanding this invention. This example is intended to be generally illustrative of this invention and is not intended to be construed as unduly limiting the reasonable scope of this invention.

The polyproylene used in this example was produced using a titanium chloride catalyst system. These types of catalyst systems are generally known in the art. The polymerization scheme to produce this polymer using this type of catalyst system is also generally known in the art. This polymer can be described as a propylene-ethylene random copolymer which has both an atactic and an isotatic molecular structure. The density of this polymer was about 0.90 grams per cubic centimeter as measured by ASTM-D-1505. The melt flow of this polymer was about 1.8 grams per 10 minutes as measured by ASTM-D-1238 at 230/2.16 Condition L.

The apparatus used in this example was essentially composed of a carbon dioxide reservoir, (hereafter, "reservoir"), a flash tank, (hereafter, "tank") a gas flow meter, (hereafter, "meter"), and an odor extraction vessel, (hereafter, "vessel"). The reservoir contained essentially pure liquid carbon dioxide. This reservoir was connected via tubing with the vessel. The vessel was a 2 inch, schedule 80 pipe vessel, with a 0.0205 square foot cross-section. It was equipped with a pressure gauge and a temperature gauge. The temperature of the vessel was controlled by a furnace which surrounded the vessel. The vessel was connected via tubing to the tank. The tank was used to collect liquid which might come from the carbon dioxide stream. The tank was connected via tubing to the meter. The meter was used to collect the carbon dioxide stream's temperature, pressure and flow rate.

The procedure used to perform the odor extraction follows. The vessel was loaded with an amount of polypropylene. The vessel was then sealed and pressure tested for leaks. High pressure, liquid carbon dioxide from the reservoir was then fed into the vessel. The extraction pressure was maintained by the use of a back pressure control valve. The temperature was controlled by the furnace. The carbon dioxide stream which exited the vessel then entered the tank which was generally maintained at a pressure of about 600 pounds per square inch. Although there was a liquid collection section in the tank, essentially no substantial portions of liquid were recovered. After running the extraction for about one hour, the vessel was allowed to cool to room temperature. The vessel was then depressurized and the polymer was removed from the vessel.

The extracted polymer was then odor tested using procedures similar to those found in the ASTM Special Technical Publication 434 entitled "Manual on Sensory Testing Methods" In general, 25 grams of each of the extracted polymers were sealed individually in a glass jar. These jars were then heated to a temperature of 90° C. This temperature was then maintained for 30 minutes and then the jars were allowed to cool to room temperature.

The odor panel then evaluated each polymer for both its odor intensity and its odor quality. The odor intensity is a measure of how strong an odor was perceived by the odor panelists while they ignored the odor quality. The odor intensity was measured on a scale of 0 to 9 where a 0 meant that the sample had essentially no odor intensity and 9 meant that the sample had a very strong odor intensity. Furthermore, the odor panel was informed that the scale was arithmetic. That is, an odor intensity of 6 would be 6 times stronger than an odor intensity of 1, and an odor intensity of 4 would be twice as strong as an odor intensity of 2. The odor quality is a measure of how pleasant or how revolting an odor was perceived by the odor panelists while they ignore the odor intensity. The odor quality is also known in the art as the hedonic tone. The odor quality was measured on a scale of −5 to +5 where −5 meant that the sample had a revolting odor and +5 meant that the sample had a pleasant odor and 0 meant that the sample had a neutral odor. Overall a sample with the lowest odor intensity value and an odor quality value closest to zero is the most preferred for commercial reasons.

The results of this experimentation are presented below in Table I.

TABLE I

Run	Column Identification Number															
Number	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
11	398.4	2205	15.30	2.07	113	318	1.05	0.10	4.72	122	323	0.4	0.104	4	−1	17
12	423.8	2205	15.30	2.07	75	297	0.98	0.10	4.72	85	303	0.7	0.106	4	−1	17
13	422.1	1700	11.82	1.60	94	308	1.01	0.10	4.72	126	325	0.8	0.107	3	0	9
14	419.3	1190	8.31	1.13	113	318	1.05	0.10	4.72	132	329	0.2	0.103	3	0	9
15	404.2	1190	8.31	1.13	75	297	0.98	0.10	4.72	119	321	0.2	0.103	3	0	9
16	423.9	2205	15.30	2.07	94	308	1.01	0.60	28.32	107	315	0.5	0.105	2	−1	5
17	418.0	1700	11.82	1.60	94	308	1.01	0.60	28.32	102	312	1.0	0.108	2	0	4
18	434.2	1700	11.82	1.60	94	308	1.01	0.60	28.32	104	313	0.6	0.106	1	1	2
19	426.3	1700	11.82	1.60	94	308	1.01	0.60	28.32	148	338	0.3	0.103	2	0	4
20	421.2	1700	11.82	1.60	75	297	0.98	0.60	28.32	111	317	0.7	0.106	3	−1	10
21	426.0	1190	8.31	1.13	94	308	1.01	0.60	28.32	143	335	0.5	0.105	2	0	4
22	414.4	2205	15.30	2.07	113	318	1.05	1.00	47.20	112	318	0.7	0.106	3	0	9
23	434.8	2205	15.30	2.07	75	297	0.98	1.00	47.20	130	328	1.0	0.108	1	0	1
24	417.0	1700	11.82	1.60	94	308	1.01	1.00	47.20	121	323	1.0	0.108	2	−1	5
25	422.9	1190	8.31	1.13	113	318	1.05	1.00	47.20	81	300	1.0	0.108	3	−1	10

TABLE I-continued

Run	Column Identification Number															
Number	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
26	421.4	1190	8.31	1.13	75	297	0.98	1.00	47.20	92	306	0.5	0.108	2	1	5

Column Identification
1. This is the mass of the polymer in grams.
2. This is the pressure measured at the odor extraction vessel in psig.
3. This is the approximate pressure at the odor extraction vessel in megapascals. Atmospheric pressure was assumed to be 0.10 megapascals.
4. This is the standard pressure. The standard pressure is equal to the pressure in the odor extraction vessel (psig + 14.696 psi) divided by the critical pressure of carbon dioxide which is 1071 psi.
5. This is the temperature measured at the odor extraction vessel in degrees Fahrenheit.
6. This is the approximate temperature in the odor extraction vessel in Kelvins.
7. This is the standard temperature. The standard temperature is equal to the temperature in the odor extraction vessel divided by the critical temperature of carbon dioxide which is 304 K. It is important to use Kelvins in calculating the standard temperature.
8. This is the gas flow rate of the carbon dioxide through the odor extraction vessel as measured at the gas flow meter in cubic feet per minute.
9. This is the approximate gas flow rate of the carbon dioxide through the odor extraction vessel in cubic meters per second $\times 10^5$.
10. This is the temperature of the carbon dioxide measured at the gas flow meter in degrees Fahrenheit.
11. This is the approximate temperature of the carbon dioxide at the gas flow meter in Kelvins.
12. This is the pressure of the carbon dioxide measured at the gas flow meter in psig.
13. This is the approximate pressure of the carbon dioxide at the gas flow meter in megapascals. Atmospheric pressure was assumed to be 0.10 megapascals.
14. This is the odor intensity score (OIS).
15. This is the odor quality score (OQS).
16. This is the odor impact score. It is equal to $OIS^2 + OQS^2$. It represents the relative impact of the odor, on an average person.

For comparison purposes two samples of unextracted polymer were also evaluated by the odor panel using the same techniques as above. Both of these samples had an odor intensity of 5 which was the highest odor intensity. The average odor quality of the samples was −1.5, (−2, −1, respectively). This represents one of the most disagreeable odors observed. The odor impact score of these polymers was 29 and 26, respectively. Overall, these two samples had an intense, disagreeable odor which would be highly objectional if used in a human consumer product mode.

However, as can be seen from Runs 11–26 subjecting this polymer to carbon dioxide at these pressures and temperatures greatly improved the odor intensity, (range = 1 to 4 point decrease, average = 2.5 point decrease), while slightly improving the odor quality, (range = 0.5 to 2.5 point decrease, average = 1.25 point decrease). Furthermore, the odor impact scores were reduced from the high twenties to almost zero. Therefore, it can be concluded that at these relatively low pressures and low temperatures polypropylene can be deodorized.

It is also interesting to note Runs 11–15. It seems that at low gas flow rates the deodorization effect seems to be independent of the temperature. This is apparent from comparing Runs 11 to 12 and Runs 14 to 15. In each case the pressure remained the same and the temperature changed, yet no odor change was observed. However, comparing Runs 11 to 14 and 12 to 15 it is apparent that while the temperature stayed the same and the pressure decreased, the odor intensity and odor quality of the polymer improved.

That which is claimed is:

1. A process to deodorize an odorous solid poly(-mono-1-olefin) said process comprising:

contacting, under deodorizing conditions, at least one deodorizing composition with at least one poly(-mono-1-olefin); wherein said deodorizing conditions comprise

a standard pressure from about 1 to about 5 and

a standard temperature from about 0.5 to about 2.5;

wherein said poly(mono-1-olefin) is substantially in the solid state.

2. A process according to claim **1** wherein said poly(-mono-1-olefin) is selected from the group consisting of homopolymers, copolymers, or a mixture of at least one homopolymer and at least one copolymer.

3. A process according to claim **2** wherein said poly(-mono-1-olefin) consists of a copolymer.

4. A process according to claim **3** wherein said copolymer consists of propylene and at least one other mono-1-olefin.

5. A process according to claim **4** wherein said mono-1-olefin consists of ethylene.

6. A process according to claim **1** wherein said poly(-mono-1-olefin) has an molecular structure selected from the group consisting of an atactic molecular structure, a syndiotactic molecular structure, an isotactic molecular structure or a mixture of two or more of said molecular structures.

7. A process according to claim **2** wherein said copolymer and said homopolymer have an molecular structure selected from the group consisting of an atactic molecular structure, a syndiotactic molecular structure, an isotactic molecular structure, or a mixture of two or more of said molecular structures.

8. A process according to claim **7** wherein said copolymer has a molecular structure consisting of a mixture of an atactic molecular structure and an isotactic molecular structure.

9. A process according to claim **8** wherein said copolymer consists of propylene and at least one other mono-1-olefin.

10. A process according to claim **9** wherein said mono-1-olefin consists of ethylene.

11. A process according to claim **7** wherein said homopolymer has a molecular structure consisting of a mixture of an atactic molecular structure and an isotactic molecular structure.

12. A process according to claim **1** wherein said mono-1-olefin in said poly(mono-1-olefin) has the following molecular structure

wherein each X is independently selected from the group consisting of hydrogen, fluorine, chlorine, bromine, and iodine, and wherein each R is selected from the group consisting of hydrogen, fluorine, chlorine, bromine, iodine, linear alkyl radicals that have from one to about thirty-two carbon atoms inclusive in their mo-

lecular structure, and branched alkyl radicals that have one to thirty-two carbon atoms inclusive in their molecular structure.

13. A process according to claim 7 wherein the mono-1-olefins in said copolymer and said homopolymer have the following molecular structure

wherein each X is independently selected from the group consisting of hydrogen, fluorine, chlorine, bromine, and iodine, and wherein each R is selected from the group consisting of hydrogen, fluorine, chlorine, bromine, iodine, linear alkyl radicals that have from one to about thirty-two carbon atoms inclusive in their molecular structure, and branched alkyl radicals that have one to thirty-two carbon atoms inclusive in their molecular structure.

14. A process according to claim 13 wherein said mono-1-olefin is selected from the group consisting of ethylene, vinyl chloride, tetrafluoroethylene, propylene, 1-butene, 3-methyl-1-butene, 1-pentene, 3-methyl-1-pentene, 4-methyl-1-pentene, 1-hexene, 3-ethyl-1-hexene, 1-heptene, 1-octene, 1-nonene, 1-decene, dodecene, tetradecene, hexadecene, and mixtures of two or more of said mono-1-olefins.

15. A process according to claim 14 wherein said mono-1-olefin consists of a mixture of said propylene and at least one other mono-1-olefin.

16. A process according to claim 15 wherein said other mono-1-olefin is ethylene.

17. A process according to claim 1 wherein said deodorizing composition is selected from the group consisting of ammonia, boron trifluoride, carbon dioxide, krypton, phosphine, chlorotrifluorosilane, silane, silicon tetrafluoride, xenon, monochlorodifluoromethane, trifluoromethane, monofluoromethane, monobromotrifluoromethane, monochlorotrifluoromethane, dichlorodifluoromethane, tetrafluoromethane, acetylene, ethylene, 1,1-difluoroethylene, ethane, dimethylether, propadiene, methyl acetylene, propylene, propane, isobutane, perfluorobutane, methanol, ethanol, propanol, water, or mixtures of two or more of said deodorizing compositions.

18. A process according to claim 13 wherein said deodorizing composition is selected from the group consisting of ammonia, boron trifluoride, carbon dioxide, krypton, phosphine, chlorotrifluorosilane, silane, silicon tetrafluoride, xenon, monochlorodifluoromethane, trifluoromethane, monofluoromethane, monobromotrifluoromethane, monochlorotrifluoromethane, dichlorodifluoromethane, tetrafluoromethane, acetylene, ethylene, 1,1-difluoroethylene, ethane, dimethylether, propadiene, methyl acetylene, propylene, propane, isobutane, perfluorobutane, methanol, ethanol, propanol, water, or mixtures of two or more of said deodorizing compositions.

19. A process according to claim 1 wherein said standard temperature is in the range of about 1 to about 4.

20. A process according to claim 18 wherein said standard temperature is in the range of about 1 to about 4.

21. A process according to claim 1 wherein said standard temperature is in the range of about 0.75 to about 1.5.

22. A process according to claim 18 wherein said standard temperature is in the range of about 0.75 to about 1.5.

23. A process according to claim 1 wherein said standard pressure is in the range of about 1 to about 4, said standard temperature is in the range of about 0.75 to about 1.5.

24. A process according to claim 18 wherein said standard pressure is in the range of about 1 to about 4, said standard temperature is in the range of about 0.75 to about 1.5.

25. A process according to claim 1 wherein said standard pressure is in the range of 1 to 3 inclusive, said standard temperature is in the range of 0.90 to 1.10 inclusive.

26. A process according to claim 18 wherein said standard pressure is in the range of 1 to 3 inclusive, said standard temperature is in the range of 0.90 to 1.10 inclusive.

27. A process according to claim 1 wherein said deodorizing composition comprises carbon dioxide.

28. A process according to claim 18 wherein said deodorizing composition consists of a mixture of carbon dioxide and at least one other deodorizing composition.

29. A process according to claim 18 wherein said deodorizing composition consists of carbon dioxide.

30. A process according to claim 1 wherein said poly(mono-1-olefin) is contacted with a metal complexing agent prior to said contacting with the deodorizing composition.

31. A process according to claim 30 wherein said metal complexing agent is selected from the group consisting of methanol, ethanol, propanol, 1,3-propanediol, 1,4-butanedione, acetylacetone, and mixtures of two or more of said metal complexing agents.

32. A process to deodorize an odorous, solid poly(mono-1-olefin) said process comprising contacting, under deodorizing conditions, carbon dioxide with said poly(mono-1-olefin), wherein said deodorizing conditions comprise a standard pressure from 1 to about 3 inclusive, and a standard temperature from 0.9 to 1.10 inclusive, and wherein said mono-1-olefin in said poly(mono-1-olefin) comprises propylene. .

33. A process according to claim 32 wherein said pressure is from 8 to 16 megapascals and said temperature is 275 Kelvins to 335 Kelvins.

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United States Patent [19]

Fodor et al.

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[54] **DEODORIZING ODOROUS POLYOLEFINS WITH LOW CONCENTRATIONS OF INORGANIC OXIDIZING AGENTS**

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[57] **ABSTRACT**

This invention relates to deodorizing odorous polyolefins. This is accomplished by mixing the odorous polyolefin with low concentration levels of an inorganic oxidant. Optionally, a fragrance is mixed with the odorous polyolefin mixture. Additionally, if desired, heat can be applied to the mixture.

16 Claims, No Drawings

DEODORIZING ODOROUS POLYOLEFINS WITH LOW CONCENTRATIONS OF INORGANIC OXIDIZING AGENTS

BACKGROUND OF THE INVENTION

This invention relates to a process for deodorizing odorous polyolefins.

Various methods are known to produce polyolefins. These polyolefin type compounds have been used in a wide variety of applications. For example, polyolefins are fabricated into molded articles such as pipes, films, and fibers. However, one persistent problem that has limited the development of polyolefin applications has been the generation of odorous compounds during the production of the polyolefin. These odorous compounds are then incorporated into the polyolefin material. After incorporation, these odorous compounds interfere with the utilization of the polyolefin material in applications where such odors would be undesirable. An example of this occurs when a polyolefin resin is used to fabricate a container for a liquid or a solid which is meant for human consumption, and that polyolefin container has a distasteful olfactory impact upon the consumer of the product.

Considering the subjectiveness of determining an odor's quality it is best if there are not any odorous compounds in the polyolefin material at all. However, it is hard, if not impossible sometimes, to determine exactly which compound, in a group of compounds, is odorous. Given the difficulties in determining which compounds are odorous, and in eliminating those odorous compounds from the polyolefin material, it is reasonable to eliminate the suspected odorous properties of the various suspected odorous compounds in order to lessen their net impact upon the final application of the polyolefin material.

SUMMARY OF THE INVENTION

It is an object of this invention to provide a process to deodorize odorous polyolefins.

It is another object of this invention to provide a process to lower the odor intensity of odorous polyolefins.

It is yet another object of this invention to neutralize the odor quality of odorous polyolefins.

In accordance with this invention, an inorganic oxidizer is mixed with the odorous polyolefin.

DETAILED DESCRIPTION OF THE INVENTION

Polyolefins

This invention is broadly applicable to polyolefins. Examples of polymerizable olefins which can be used to produce these polyolefins include, but are not limited to, those olefins which contain from 2 to 30 carbon atoms per molecule. However, more preferably, these olefins contain from 2 to 20 carbon atoms per molecule. The molecular structure of these polymerizable olefins can also be either linear or branched. Additionally, these polymerizable olefins can be polymerized alone to give a homopolymer or they can be polymerized with another monomer to give a copolymer. Individual molecular examples of polymerizable olefins include, but are not limited to, ethylene, propylene, butene, pentene, hexene, heptene, octene, 3-methyl-1-butene, 3-methyl-1pentene, 4-methyl-1-pentene, 4-methyl-1-hexene, 3-ethyl-1-hexene, 3,3-dimethyl-1-butene, 4,4-dimethyl-1-hexene, decene, dodecene, tetradecene, hexadecene, conjugated and/or non-conjugated diolefins such as 1,3-butadiene, isoprene, piperylene, 2,3-dimethyl-1,3-butadiene, 1,4-pentadiene, 1,7-hexadiene.

Inorganic Oxidizing Agents

In general, the term "inorganic oxidizing agent" refers to those non-carbon containing compounds which tend to be electron acceptors in oxidation-reduction type reactions. The term includes, but it not limited to, such classes of chemicals as inorganic peroxides, chlorates, perchlorates, nitrates, and permanganates. These types of oxidizing agents can react vigorously at ambient temperatures when stored near or in contact with reducible materials such as formaldehyde and other organic compounds.

Individual examples of the types of inorganic oxidizing agents that can be utilized are:

1. Permanganates: typical examples include potassium permanganate and sodium permanganate. Although permanganates can be used as an inorganic oxidizing agent, they tend as a group to impart an unacceptable color to the polyolefin. Thus they are not as useful as the other classes;

2. Nitrates: typical examples of nitrates include calcium nitrate and potassium nitrate;

3. Perchlorates: typical examples include ammonium perchlorate, potassium perchlorate and sodium perchlorate. Although there are many perchlorates in all of the groups of the periodic table, perchlorates of group IA and group IIA are preferred over the other perchlorates;

4. Chlorates: typical examples include sodium chlorate and potassium chlorate;

5. Peroxides: typical examples include hydrogen peroxide, sodium peroxide, sodium peroxyborate.

Other examples include such compounds as sodium hypochlorite and potassium persulfate. Of these types of compounds hydrogen peroxide is most preferred due to its availability, reactivity and lack of undesireable residuals left in the polymer after treatment.

Hydrogen Peroxide

The hydrogen peroxide used in this invention can be of any concentration. The required hydrogen peroxide can be obtained in concentrations ranging from about 3 to about 98 percent by weight based on the total weight of peroxide and water. However, the preferred concentration of hydrogen peroxide to use is from about 3 to about 70 percent by weight. Even more preferably a concentration of hydrogen peroxide is from about 3 to about 30 percent by weight. Concentrations above about 70 weight percent promote organic oxidation and require special handling procedures. Concentrations below about 3 weight percent are expensive to stabilize and transport thus increasing processing costs.

Amount of Inorganic Oxidizing Agent to Use

The amount of inorganic oxidizing agent to mix with the odorous polyolefin is preferably from about 0.0001 to about 0.1 weight percent based on the weight of the polyolefin. However more preferably, the inorganic oxidizing agent is from about 0.0005 to about 0.05 weight percent, and most preferably from about 0.001 to about 0.02 weight percent, where all the weight percents are based on the weight of the polyolefin.

If too much inorganic oxidizing agent is added this will promote undesirable side effects such as cross-linking and vis-breaking in the polyolefin material. If too little inorganic oxidizing agent is added the odor intensity and odor quality of the odorous polyolefin might be unaffected. Preferably, the amount added lowers the odor intensity and neutralizes the odor quality of the odorous polyolefin.

It should be noted that inorganic oxidizing agents can present a serious safety hazard. Great care should be taken when handling these compounds especially when these compounds are in contact with organic materials.

Fragrance

Optionally, a fragrance may be mixed with the inorganic oxidizing agent and odorous polyolefin. The type of fragrance to add depends on the particular properties of the fragrance and the end use of the polyolefin. Some types of fragrances to add are, for example, lemon oil, lime oil, mandarin oil, verbena, and lemon grass oil.

The amount of fragrance to mix with the odorous polyolefin is preferably from about 0.0025 to about 0.1 weight percent based on the weight of the polyolefin. However, more preferably the amount of fragrance to add is from about 0.005 to about 0.075 weight percent and most preferably, it is from about 0.0075 to about 0.05 weight percent, where all the weight percents are based on the weight of the polyolefin.

If too much fragrance is added the fragrance will adversely odorize the odorous polyolefin resulting in undesirable olfactory impact upon the end user of the polyolefin. If too little fragrance is added the odor intensity and odor quality will not be beneficially changed. Preferably, the amount added lowers the odor intensity and neutralizes the odor quality of the odorous polyolefin.

Heat

Optionally, heat may be applied to the polyolefin mixture that contains the inorganic oxidizing agent and optionally a fragrance. The heat can be applied by any known method in the art. The amount of heat to be applied depends on the type of polyolefin that is to be deodorized. Generally, the temperatures at which the polyolefin is deodorized is below the melting point of that polyolefin. For example, polypropylene should be deodorized below its melting point of about 160°C. If the temperature at which the polyolefin is deodorized is too high this could produce undesirable side effects such as structural degradation. If the temperature at which the polyolefin is deodorized is too low the process will proceed too slowly to be economically useful.

Reaction Conditions

The inorganic oxidizing agent can be mixed with the polyolefin at any time after its formation and removal from the reactor. However, this process should be done before the polyolefin is finally processed. For example, this process could be accomplished after the polyolefin is formed, but before the polyolefin is passed through an extruder. As a further example, the polyolefin after its removal from the reactor, could be placed in a mixer where an inorganic oxidizer is added. Optionally, at this time, a fragrance can also be added. The resulting mixture could then, if desired, be passed through a Double Agitator Heat Treater where frictional heat could be applied. Additionally, as another example, the polyolefin after its removal from the reactor, could be passed through a purge conveyor where an inorganic oxidizer is mixed with the polyolefin. Additionally, a fragrance can be added, as well as heat applied to the polyolefin mixture as it passes through the purge conveyor. It should be noted that it is known in the art to protect a polyolefin by the addition of antioxidants to the polyolefin material. Therefore, this invention would seem to run in opposition to accepted practice. While not wanting to be bound by theory, it is believed that the addition of the inorganic oxidizing agent decreases the odor intensity and neutralizes the odor quality of the polyolefin by preferentially attacking the odorous compounds while essentially leaving the antioxidant protected polyolefin unharmed.

This process can be done under pressure with an upper pressure limit of about 10,000 psi. However, near atmospheric pressure is preferred for ease of processing and for safety reasons.

The polyolefin can be left in contact with the inorganic oxidizer, for about five seconds to about five hours. However, more preferably, the polyolefin is left in contact for about one minute to about 3 hours. If the polyolefin is allowed contact with the inorganic oxidizer for too long, undesirable side effects may occur. For example, the process might generate free radicals in unwanted amounts causing damage to the polyolefin. If the polyolefin is not allowed sufficient contact time with the process then the polyolefin will not be sufficiently deodorized. Additionally, if heat is applied to the polyolefin for too long of a time structural degradation could occur.

This process can be accomplished under different types of media. For example, the process can be carried out in an inert atmosphere, a nitrogen atmosphere, or any normal atmosphere. However, the atmosphere chosen should not be highly reactive with the inorganic oxidizer in order to lessen the risk of a safety hazard.

As a further help in understanding the present invention and its advantages the following example is provided.

EXAMPLE

A Dynamic Dilution Binary Scale Olfactometer (DDBSO) was used to reference the odor intensity of the samples. This referencing device is similar to that described in ASTM E544-75 (reapproved 1988). This ASTM is a standard for referencing suprathreshold odor intensity. However, this standard only references odor intensity and not odor quality.

The DDBSO provided reference data by continuously and simultaneously preparing an air/odor vapor mixture at eight different concentration levels. These concentration levels followed a geometric progression with each succeeding level being a factor of 2 greater than its preceding level. For example, the concentration level at 4 was about twice as much as the concentration level at 3, yet level 4 had half as much as the concentration level at 5. This measure of the odor intensity was a measure of how strong the odor was perceived by the odor panelists while they ignored the odor quality. The odor panelists responded on a scale of 1 to 8 where 1 represented the lowest odor intensity and 8 represented the strongest odor intensity.

The odor quality (also referred to in the art as the hedonic tone) was a measure of how pleasant or how revolting a particular odor was perceived. This odor quality was measured on a −3 to a +3 scale where −3 represented a revolting odor and +3 represented a

pleasant odor. The 0 point on this scale represented a neutral odor quality. For commercial reasons a neutral odor quality is usually desirable for a polyolefin product.

The reactants used in this example were hydrogen peroxide, Naarden lemon oil, IFF "Golden Forest", and polypropylene. The hydrogen peroxide used was an aqueous 30 weight percent solution which is commercially available. The Naarden lemon oil is commercially available on the market. IFF "Golden Forest" is a fragrance from the International Fragrance and Flavor Company. The polypropylene was in the form of powder. This powder was made by methods known in the art. After treatment, this polypropylene powder was then formed into pellets or into blow molded bottles to be odor sampled. The device used to apply heat to the process was a Double Agitator Heat Treater also known in the art as a Wedco polisher. This device imparts heat to the odorous polyolefin mixture by frictional means. The device employs no external heat source and no heat transfer media. Essentially, all power delivered from the drive motor to the agitator is transferred directly to the material thus providing frictional heating to the material. The Double Agitator Heat Treater was operated at a temperature of 115° C.

Preparation of the Samples

The untreated samples (runs 01 and 11) were prepared by taking untreated polypropylene powder and forming it into pellets (run 01) or into a blow molded bottle (run 11).

Runs 02 through 10 were prepared by adding the listed embodiments and then forming the polypropylene into pellets. For example in run 04, 0.01 weight percent of lemon oil and 0.01 weight percent of hydrogen peroxide were mixed with untreated polyproylene powder. Next this mixture was passed through the Double Agitator Heat Treater where the heat was added. Finally, the treated polypropylene was pelleted for sampling.

Runs 12 through 21 were prepared the same way runs 02 through 10 were prepared except that they were further formed into blow molded bottles.

Column Explanation

Column A represents the type of sample used. In this column "Pellet" represents a polypropylene pellet sample, while a "Bottle" represents a polypropylene blow-molded bottle sample. These two types of samples were selected because:

1) the pellet samples give a good indication of the odor of the polypropylene as it leaves the plant site; and

2) the blow-molded bottles give a reasonable indication of the odor of the polypropylene after being shaped into its end use form. However, this data would be greatly affected by the type of shaping method used by the end product shaper. The reason is that these shaping methods can impart additional odors to the polypropylene.

Additionally, the blow-mold bottle samples were heated for two hours at 60° C. then cooled to room temperature before sampling.

Column B indicates how much 30% aqueous hydrogen peroxide was added. This figure is the weight percent of hydrogen peroxide where the weight percent of the hydrogen peroxide is based on the weight of the polypropylene.

Column C indicates whether a Wedco polisher was used on the sample. A "YES" indicates that the sample was run through the Wedco polisher and an "NO" indicates that the sample was not run through the Wedco polisher.

Column D indicates how much Naarden lemon oil was added. This figure is the weight percent of lemon oil where the weight percent of the lemon oil is based on the weight of the polypropylene.

Column E represents how much IFF "Golden Forest" was added. This figure is the weight percent of IFF "Golden Forest" where the weight percent of IFF "Golden Forest" is based on the weight of the polypropylene. IFF "Golden Forest" is based on the weight of the polypropylene. IFF "Golden Forest" stands for the International Fragrance and Flavors Company's fragrance called "Golden Forest."

Column F represents the average of the odor panelists' reaction to the odor intensity. This was measured on a scale of 1–8 where 1 represented the lowest intensity and 8 represented the strongest intensity. Additionally, each increment of one represents a factor of two increase or decrease in the odor intensity. For example, in run number 01 the odor intensity was 5.3 whereas, in run number 04 the odor intensity was 3.5, this change in the odor intensity of 1.8 units (5.3 to 3.5) represents a nearly four fold decrease in the odor intensity of the sample.

Column G represents the average of the odor panelists' reactions to the odor quality. This was measured on a scale of −3 to +3, where −3 represented a revolting odor, zero represented a neutral odor, and +3 represented a pleasant odor.

Column H represents the range of responses given by the odor panelists when they sampled the odorous polypropylene. This was measured using the same scale as in column G. For example, in run number 01 several odor panelists thought that the untreated polypropylene pellets had an unpleasant odor, while others thought that the untreated polypropylene had a neutral odor. Whereas, in run number 04 the odor panelists thought that the treated polypropylene had either a neutral odor or a pleasant odor.

Column I represents the net change in the odor quality of the treated polypropylene sample when compared to the untreated polypropylene sample. In other words, each odor panelists' perception of the treated polypropylene was compared to their perception of the untreated polypropylene. The change in these perceptions is the data that is recorded in column I. For example, in run number 21 some odor panelists definitely thought that the addition of Naarden lemon oil worsened the odor while others thought that this addition improved the odor when compared to the untreated blow molded bottle sample. In contrast, in run number 04 the odor panelists thought that the added constituents either did not change the odor quality or thought the odor was greatly improved when compared to the untreated pellet sample. Additionally, each treated sample was only compared with the similar type untreated sample. In other words, samples of treated polypropylene pellets (runs 02 through 10) were compared with the untreated polypropylene pellets (run 01), whereas samples of treated polypropylene blow molded bottles (runs 12 through 21) were compared with the untreated polypropylene blow molded bottle (run 11).

RUN NUMBER	A TYPE OF SAMPLE	B HYDROGEN PEROXIDE	C HEAT	D LEMON OIL	E IFFGF	F ODOR INTENSITY	G VALUE	H ODOR QUALITY RANGE	I CHANGE
01	PELLET	0.00	NO	0.00	0.0000	5.3	−1.3	−2 → 0	N/A
02	PELLET	0.01	NO	0.00	0.0000	5.3	−0.5	−2 → +1	0 → +2
03	PELLET	0.01	YES	0.00	0.0000	4.0	0	−1 → +1	0 → +3
04	PELLET	0.01	YES	0.01	0.0000	3.5	+0.5	0 → +2	0 → +4
05	PELLET	0.01	YES	0.00	0.0025	5.3	+1	0	+1 → +3
06	PELLET	0.01	NO	0.01	0.0000	5.0	0	−1 → +1	0 → +3
07	PELLET	0.01	NO	0.00	0.0025	5.3	+0.3	−1 → +1	0 → +3
08	PELLET	0.00	YES	0.00	0.0000	4.3	−1	−3 → 0	−1 → +1
09	PELLET	0.00	YES	0.01	0.0000	4.5	+1.3	−1 → +3	0 → +4
10	PELLET	0.00	YES	0.00	0.0025	4.5	+0.8	−1 → +2	−1 → +4
11	BOTTLE	0.00	NO	0.00	0.0000	5.3	−1	−2 → 0	N/A
12	BOTTLE	0.01	NO	0.00	0.0000	5.3	−0.8	−2 → 0	−1 → +1
13	BOTTLE	0.01	YES	0.00	0.0000	4.3	−0.5	−1 → 0	−1 → +2
14	BOTTLE	0.01	YES	0.01	0.0000	4.5	−0.8	−2 → +1	−1 → +2
15	BOTTLE	0.01	YES	0.00	0.0025	4.3	−1	−2 → 0	−1 → +1
16	BOTTLE	0.01	NO	0.01	0.0000	4.5	−0.5	−1 → +1	−1 → +2
17	BOTTLE	0.01	NO	0.00	0.0025	3.8	−1	−2 → +1	−1 → +2
18	BOTTLE	0.00	YES	0.00	0.0000	6.0	−1.8	−3 → −1	−3 → 0
19	BOTTLE	0.00	YES	0.01	0.0000	4.0	−0.3	−1 → +1	−1 → +2
20	BOTTLE	0.00	YES	0.00	0.0025	4.3	−0.5	−1 → +1	−1 → +2
21	BOTTLE	0.00	NO	0.01	0.0000	3.8	−0.8	−3 → +1	−2 → +2

From the above data it is apparent that the odor intensity was improved the best by run number 04. This run which included the optional constituents and showed an odor intensity improvement of 34% (5.3 to 3.5) on the binary geometric progression odor intensity scale. Additionally, it is apparent that the odor quality, as reported by the odor panelists, was improved from −1.3 to 0.5 on the odor quality scale. Furthermore, run 04 showed that the run had one of the greatest beneficial impacts on the odorous polypropylene with the odor panelists reporting that either they did not change their perception of the odorous polypropylene (a score of 0) or that their perception was changed greatly (a score of +4). And lastly, run 04 was the only run the odor panelists reported that the odor quality was either neutral or pleasant in their perception.

Additionally, it should be noted that all of the runs using the peroxide showed improvement due to the peroxide alone. For example, a comparison of runs 01 and 02 shows an improvement in the odor quality. A comparison of runs 03 and 08 shows that the addition of the peroxide to the heat treated sample improved the odor intensity and the odor quality. Similarly in runs 11 and 12 an improvement in the odor quality was reported. Furthermore, a comparison of 13 and 18 showed an improvement in both the odor quality and odor intensity.

While this invention has been described in detail for the purpose of illustration it is not to be construed as limited thereby, but is intended to cover all changes and modifications within the spirit and scope thereof.

That which is claimed is:

1. A process for deodorizing odorous aliphatic polyolefins which comprises:

(a) contacting about 0.0001 weight percent to about 0.1 weight percent of inorganic oxidizing agent selected from the group consisting of ammonium perchlorate, potassium perchlorate, sodium perchlorate, sodium chlorate, potassium chlorate, sodium peroxide, sodium peroxyborate, hydrogen peroxide, and mixtures thereof, where the weight percent of inorganic oxidizing agent is based upon the weight of the aliphatic polyolefin; with

(b) said odorous aliphatic polyolefin;

to reduce the odor intensity and neutralize the odor quality of said aliphatic polyolefin.

2. A process according to claim 1 wherein said inorganic oxidizing agent is hydrogen peroxide.

3. A process according to claim 1 further comprising contacting about 0.0025 weight percent to about 0.1 weight percent of fragrance where the weight percent of fragrance is based upon the weight of the aliphatic polyolefin; with said inorganic oxidizing agent and said odorous aliphatic polyolefin to reduce the odor intensity and neutralize the odor quality of the polyolefin.

4. A process according to claim 3 wherein the fragrance is selected from the group consisting of lemon oil, lime oil, mandarin oil, verbena, lemon-grass oil, and mixtures thereof.

5. A process according to claim 3 wherein the fragrance is lemon oil.

6. A process according to claim 1 further comprising heating the polyolefin and inorganic oxidizing agent mixture to a temperature sufficient to reduce the odor intensity and neutralize the odor quality of the polyolefin.

7. A process according to claim 6 where the temperature is below the melting point of the polyolefin.

8. A process according to claim 3 further comprising heating the polyolefin, inorganic oxidizing agent, and fragrance mixture to a temperature sufficient to reduce the odor intensity and neutralize the odor quality of the polyolefin.

9. A process according to claim 8 where the temperature is below the melting point of the polyolefin.

10. A process for deodorizing odorous polypropylene which comprises:

(a) mixing with the odorous polypropylene, about 0.0001 weight percent to about 0.1 weight percent of hydrogen peroxide, where the weight percent of hydrogen peroxide is based upon the weight of the polypropylene;

(b) mixing with the odorous polypropylene, about 0.0025 weight percent to about 0.1 weight percent of lemon oil where the weight percent of lemon oil is based upon the weight of the polypropylene; and

(c) heating the odorous polypropylene, hydrogen peroxide, and lemon oil mixture to a temperature

below the melting point of the polypropylene to reduce the odor intensity and neutralize the odor quality of the odorous polypropylene.

11. A process for deodorizing aliphatic polyolefins which comprises:

(a) forming a powdered aliphatic polyolefin product;

(b) contacting said powdered aliphatic polyolefin product with an antioxident to form an antioxident protected polyolefin;

(c) contacting said antioxident protected polyolefin with about 0.0001 to about 0.1 weight percent of inorganic oxidizing agent where the weight percent of inorganic oxidizing agent is based upon the weight of said powdered aliphatic polyolefin product; and

(d) extruding the product of step (c).

12. A process according to claim **11** wherein said contacting step (c) occurs for about 5 seconds to about 5 hours.

13. A process according to claim **11** wherein step (c) further comprising heating at a temperature below the melting point of said powdered aliphatic polyolefin product.

14. A process according to claim **11** wherein step (c) further comprises contacting with about 0.0025 to about 0.1 weight percent of fragrance wherein the weight percent of fragrance is based on the weight of said powdered aliphatic polyolefin product.

15. A process according to claim **14** wherein the fragrance is selected from the group consisting of lemon oil, lime oil, mandarin oil, verbena, lemon-grass oil, and mixtures thereof.

16. A process according to claim **14** wherein the fragrance is lemon oil.

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